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**IDA DOCUMENT D-1454** 

GRAPHITE-REINFORCED POLYCYANATE COMPOSITES FOR SPACE AND MISSILE APPLICATIONS

Janet M. Sater Michael A. Rigdon



November 1993

Prepared for Ballistic Missile Defense Organization



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INSTITUTE FOR DEFENSE ANALYSES

Contract MDA 903 89 C 0003 Task T-R2.597.09

#### **PREFACE**

LtCol Michael Obal of the Ballistic Missile Defense Organization, Materials and Structures Office, manages a wide variety of advanced technology and demonstration programs addressing needs for various systems. A number of demonstration programs have been initiated over the past few years. These projects include fabrication and test of adaptive structures for jitter control and sensor system performance, and lightweight spacecraft and missile structures. Advanced polymer composites have been selected for fabrication of many of these structures. Increasing interest in graphite-reinforced polycyanate composites by the prime contractors raised some questions regarding the status of this material in terms of its availability, processability, data, and fabrication and test experience. A workshop involving structural designers, material vendors, and parts fabricators was planned, in part, to promote communication among the appropriate groups. The agenda was put together to identify technical issues and limitations associated with graphite-reinforced polycyanate composites.

The workshop was hosted by IDA on June 16, 1993. IDA was requested under BMDO Task T-R2-597.09 to participate in the workshop and to prepare a proceedings to document the content of the workshop. This effort was subsequently carried out by Dr. Janet Sater with input from LtCol Michael Obal and Dr. Michael Rigdon (IDA).

## **ABSTRACT**

LtCol Michael of the BMDO Materials & Structures sponsored this workshop to promote communications among the participating groups and to identify technical issues and limitations associated with graphite-reinforced polycyanate composites. Some of the identified issues include a lack of standard materials and associated process specifications due to continuing changes in resin and prepreg formulations. Perhaps the most discussed issue was the lack of a statistical database, due, in part, to the changing formulations just mentioned. Development of a handbook via government coordination of existing information may be a way to provide data to designers and increase material acceptance. Additional topics discussed were high resin contents in prepreg tapes; the lack of appropriate protective coatings; a need for structural bonding processes; a need for additional, environment-related data, particularly effects on mechanical/physical properties; a need for joint tests and subsystem level tests of structural components; and limited design tools and standards for using composites.

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## **GLOSSARY**

ACESA Advanced Composites with Embedded Sensors and Actuators

ACESS All Composite Experimental Spacecraft Structure

ACTEX Advanced Control Technology Experiment

ACTS Advanced Communications Satellite

AMASS Advanced Materials Applications for Space Structures

ASTM American Society for Testing and Materials

AXAF Advanced X-ray Astronomical Facility
BMDO Ballistic Missile Defense Organization

C/C carbon-carbon

CME coefficient of moisture expansion

COI Composite Optics, Inc.

CTE coefficient of thermal expansion

DFARS Defense Federal Acquisition Regulation Supplement

EMI electro-magnetic interference

EOIM Evaluation of Oxygen Interactions with Materials

EOS Earth Observing System
ESD electro-static discharge
GBI ground-based interceptor

Gr/EP graphite/epoxy

Gr/PC graphite-reinforced polycyanate
Gr/TP graphite-reinforced thermoplastic

Gr/TS graphite/thermoset

GSTS Ground-Based Surveillance and Tracking System

HARD High Accuracy Reflector Development

ICI Imperial Chemical Industries

IRAD Independent Research and Development

JPL Jet Propulsion Laboratory

LDCE Limited Duration Candidate Materials Exposure

LEO low Earth orbit

M&S Materials and Structures

MILSTAR Military Strategic Tactical and Relay (Satellite)

MM Martin Marietta

NASA National Aeronautics and Space Administration

NDE nondestructive evaluation

NPBSE Neutral Particle Beam Space Experiment

OSC Orbital Sciences Corporation

PAN poly-acrylo-nitrile

PC polycyanate

PL Phillips Laboratory

SAWAFE Satellite Attack Warning and Assessment Flight Experiment

SDIO Strategic Defense Initiative Organization

SPO Special Program Office
SS/L Space Systems/Loral
SSTO Single Stage to Orbit

STEP-3 Space Test Experiment Platform
STRV Space Test Research Vehicle
Tg glass transition temperature

THAAD Theater High Altitude Area Defense

TVC thrust/vector control
VEM visco-elastic material

#### 1. INTRODUCTION

LtCol Michael Obal of the Ballistic Missile Defense Organization (BMDO), Materials and Structures (M&S) Office, manages a wide variety of advanced technology and demonstration programs addressing needs for various systems. Included among the demonstration programs are fabrication and test of (1) adaptive structures for jitter control and improved sensor system performance and (2) lightweight spacecraft and missile structures. Advanced polymer composites have been selected for fabrication of many of these structures. The prime contractors have expressed an increasing interest in graphite-reinforced polycyanate composites. In response, LtCol Obal requested that IDA organize a workshop addressing material availability, processability, quality, data, and fabrication and test experience. This workshop involved structural designers, material vendors, and parts fabricators and served as a forum to promote communication among the appropriate groups and to identify technical issues and limitations associated with this material (Appendix A).

The document follows the order of the agenda. An agenda and list of attendees are contained in Appendix A. Summaries and discussion of the designer perspectives are in Chapter 2, material vendor perspectives in Chapter 3, and parts fabricator perspectives in Chapter 4. Appendixes B through D contain copies of the charts for Chapters 1 through 4, respectively. The final summary and conclusions are found in Chapter 5.

#### A. LtCol MICHAEL OBAL, BMDO

LtCol Mike Obal presented an overview of the M&S Program (p. B-3). The M&S Program has evolved through the years by taking technologies from the laboratory through proof-of-concept, component tests, subsystem and brassboard demonstrations, and full-scale ground and flight tests, to promote insertion into defense systems. The M&S charter—to reduce risk, enhance capabilities, and increase affordability in emerging materials and structural technology areas—is implemented by identifying issues via interactions with Special Program Offices (SPOs) and program managers as well as prime contractors (p. B-6). Programs are then initiated to resolve the identified issue(s). Advanced technologies being developed and demonstrated in these programs include, for example, jitter control and improved sensor system performance via adaptive structures

technology and lightweight spacecraft and missiles via advanced, graphite fiber-reinforced composites and low-cost manufacturing technology.

Early efforts in the lightweight structures technology (p. B-7) were concerned with development and coupon level testing of thermoplastic and metal matrix composites. Later programs demonstrated fabrication and testing of advanced materials in large interceptor structures, various spacecraft parts such as solar array panels, an interstage, and a hardened radiator. On-going system insertion demonstration efforts include the all-composite experimental satellite structure, a ground-based interceptor structure, and an interceptor structural technology flight demonstration. Low-cost manufacturing techniques including textile weaving, matched metal molding, and alternative structural designs have been examined for the fabrication of some of these structures (p. B-8).

LtCol Obal highlighted the Clementine structures (p. B-9) and the All-Composite Spacecraft (p. B-10). The solar array panels were fabricated for Clementine from IM7-reinforced polycyanate composites; the estimated weight savings over the Al baseline was 11 lb. The interstage structure was also fabricated for Clementine but from T650/epoxy composites; the estimated weight savings over the Al baseline was 15 lb. The all-composite spacecraft effort is a joint Boeing-Phillips Laboratory program to examine low-cost, lightweight, producible satellite designs fabricated from high stiffness, fiber-reinforced composites. The preliminary design consists of a 6-sided spacecraft with shear panels; there are two hexagonal panels to close the box.

LtCol Obal described a sensory structures project to demonstrate threat detection capabilities with minimum weight penalty to the spacecraft via attachment of various sensors to its skin as shown on page B-11. Later designs will integrate the sensors, analog-to-digital converter, and the processor into the skin (p. B-11). Future efforts along these lines may involve integration of miniaturized avionics packages or other electronic subcomponents into load-bearing structures. The schematic on page B-12 shows the integration of the control avionics and transceiver avionics into structural, load-bearing panels in a spacecraft.

LtCol Obal concluded his presentation by emphasizing the major objectives of the workshop: to promote communication among the appropriate groups and to identify technical issues and limitations associated with graphite-reinforced polycyanate composites.

#### B. DR. JANET M. SATER, IDA

Dr. Sater discussed the evolution of advanced composites in the M&S Program within the context of architecture changes in the former Strategic Defense Initiative Organization (p. B-15). The initial focus in the nonacquisition SDIO program was on thousands of interceptors and missiles such as Space-Based Interceptors, Endo-Exoatmospheric Interceptors, and very large space structures (e.g., Neutral Particle Beam, Space-Based Laser, and Boost Surveillance and Tracking System). The current BMDO program is focused on acquisition of perhaps hundreds of small interceptors and missiles (e.g., Ground-Based Interceptors and Theater Missile Defense systems) and moderate-to small-sized space systems (e.g., Brilliant Eyes and Ground-Based Radar). Important attributes of structures used in these systems include weight, mechanical and thermal properties, natural and threat survivability, and cost. In the early years of the strategic defense program there were few materials available capable of meeting the stringent system requirements may be somewhat less stringent but cost has become roughly equivalent in importance to performance.

The M&S program initially focused on material evaluation at the coupon level and on fabrication of prototype components and structures. Candidate materials included carbon-carbon (C/C), and various metal and polymer matrix composites.<sup>1</sup> These materials were selected to address particular features of the early performance requirements (pp. B-17 – B-18). For example, C/C was selected for threat survivability. As another example, the fiber-reinforced thermoplastics were selected for their high specific strength and modulus, low thermal expansion and outgassing, improved toughness/damping, and microcracking resistance over graphite/thermosets, and, most importantly, producibility and processability. A number of components and structures have been fabricated: tubes, struts, frames, and panels; joints; seeker structures, interceptor structures, spacecraft bus structures, and trusses; and, more recently, adaptive structures.

Few of the evaluated materials were transitioned to the prime contractors for insertion into real systems. The following issues, identified by Dr. Sater, apply generically to all the materials and specifically to the graphite/thermoplastics (p. B-20).

Metal matrix composites include graphite fiber-reinforced aluminum and magnesium (Gr/Al, Gr/Mg) and silicon carbide particulate- or whisker-reinforced aluminum (SiC/Al). Polymer composites include graphite-reinforced thermosets and thermoplastics (Gr/TS, Gr/TP).

- 1. The constantly changing system architecture made it difficult to perform realistic trade studies.
- 2. Material quality is an issue that pervades the advanced materials field, uniformity and reproducibility being particularly notorious problems. Residual stresses occurring as a result of fabrication processes were also noted.
- 3. The lack of the appropriate capital equipment at the prime contractors' facilities was a particular problem for the Gr/TPs: one of the advantages claimed by the thermoplastics community is the use of nonautoclave processing; the prime contractors were unwilling to purchase new, expensive processing/fabrication equipment when they already had autoclaves, a fact that made the thermoplastic composite components more expensive than the competing graphite/epoxy components.

These issues led, in part, to an interest in the graphite-reinforced polycyanate (Gr/PC) composites.

Glass- or aramid-reinforced polycyanate composites have been utilized extensively in the circuit board industry for their low dielectric properties and good peel strength (p. B-21). Other attractive features include the following: composites fabricated using a number of different processes exhibit the same properties; good translation of mechanical properties has been observed using various fibers; with the addition of toughening agents the PC composites can be tougher than epoxy composites; and they exhibit reduced moisture absorption and outgassing compared to epoxy composites. The fact that polycyanate resins process similar to epoxy resins implies that existing capital equipment can be utilized. A low cure shrinkage of <1 percent promotes resistance to microcracking from thermal cycling and provides some inherent level of toughness.

To address the objectives outlined by LtCol Obal requires the participation of several groups of people: spacecraft structural designers, material vendors, and composite parts fabricators. Dr. Sater concluded her presentation by reviewing the subject areas to be addressed by each group. Designers were asked to describe requirements/data for the application of advanced materials to real systems (primary and secondary structure) including necessary properties, qualification tests, etc.; other issues that need to be addressed for such application; and specific fiber/matrix combinations of potential interest. Material suppliers were asked to discuss information they need from designers to insure adequate material development; current availability of data including material options, material availability/maturity, density, physical and mechanical properties, outgassing, microcracking, thermal cycling effects, natural space environment survivability;

manufacturability/producibility and joining issues; estimated current and projected costs; and other potential risks. Composite parts fabricators were asked to discuss their experiences with graphite-reinforced polycyanate composites, particularly material uniformity and processing-related issues; actual components that have been fabricated, noting any difficulties; and results/data from any additional testing.

## 2. DESIGNER PERSPECTIVES

Designers were asked to define requirements and data they find necessary for advanced materials to be applied in a real system (primary and secondary structure) including properties, qualification tests, etc. They were also asked to identify other issues that need to be addressed for such application as well as specific fiber-matrix combinations that may be of interest. Other discussion topics were to include suggested R&D directions and initiatives to meet the stated needs and requirements.

#### A. DR. MOHAN ASWANI, THE AEROSPACE CORPORATION

Dr. Aswani discussed the Aerospace perspective on materials selection and material and component qualification for real systems. He began by stating that many of the advanced composites were a "solution looking for a problem," rather than the reverse. Typically a material is considered highly promising based on coupon data. For insertion into a system, however, other data are necessary. LtCol Obal commented that BMDO can not afford to provide an entire database for any material. The M&S goal is to accelerate the material development process so that industry will use it.

Material selection for a design (p. C-3) begins with a flow down of requirements such as subsystem or component weight, stiffness/strength and thermal conductivity, dimensional stability, interfaces with other subsystems, and the storage/operational environment. Candidate materials are selected based on several factors, the two most important being processability and previous application experience. Trade studies can then be performed based on certain selection criteria. In the trade studies one may investigate design flexibility—is it a material substitution or can a new design be considered? Credible models should be used and knowledge of critical parameters is essential. Schedule and cost must be factored into the trade studies.

Dr. Aswani then described steps to be avoided (p. C-4). Requirements should not be based on inadequate performance margins, unrealistic weight allocations, or unproven

According to Dr. Aswani, the entire database is not required. However, material development must be at an adequately mature level to provide confidence to a program manager.

assurances of high performance. The weight allocation problem has continually plagued spacecraft designers: it "never happens that original weight goals are met." Weight growth up to 30-40 percent (of the initial estimate) is not uncommon. Materials should not be selected based on limited property data<sup>2</sup> and experience, unreliable supply sources, and uncertain cost and schedule factors. Trade study parameters are typically weighted from 1 to 5. It is possible to manipulate them to obtain some predetermined outcome. Dr. Aswani cautioned against preselecting the material and suggested that a blind approach be used. He also warned that credible analyses must be used and subsystem interactions must be considered.

Material and component qualification are often schedule driven. No short cuts (in analyses, tests, system impact evaluation) should be taken in order to meet a schedule. As an example, it was necessary to select a material that could meet the tight delivery schedule and result in significant weight savings for the Clementine spacecraft. For a concurrent engineering approach, system level impact studies are even more critical. In addition to a balanced program of analyses and tests it is important that variability in material properties and processing be quantified. Test and inspection methods must also be proven. LtCol Obal commented that material suppliers need guidance on properties from the designer and any specifications must be written down, an example being guidance for piezoceramic material/actuator suppliers.<sup>3</sup> A typical qualification plan is illustrated on page C-6. One of the main difficulties is the desire by materials scientists to fill up the first box (materials properties tests) because some "n" number of tests are required before a designer will select a material. LtCol Obal asked if there was "a more sensible, optimal way to design" a qualification plan to address a subset of properties rather than everything. Ms. Pam Burns (Hughes) remarked that typically a company begins to develop a database for a given material, then a supplier introduces a new and/or improved version to take its place. The result is that a material is never taken to completion. It would be helpful to know the incremental benefit of the improvements in quantitative terms and to evaluate whether or not said improvements make sense, an effort that will require discipline on the part of the supplier as well as the materials research community. Mr. Mike Dean stated that

If only coupon data are available an appropriate knock-down factor should be applied to the properties utilized in the trade studies.

A workshop was held at IDA in February 1992 addressing the needs of spacecraft designers for improved piezoceramic actuator materials. A preliminary specification is to be developed. See SDIO Workshop on Piezoelectric Ceramic Actuators for Space Applications, IDA Document D-1189, June 1992, 283 pages.

Ball Aerospace uses a standard set of tests to evaluate composite material properties. Dr. Aswani indicated that there will be "different considerations for different applications." Adequate models and a good understanding of material behavior are, therefore, necessary. Ms. Burns commented that graphite/epoxy (Gr/Ep) composites have existed for years. Since there is an experience base it should not be necessary to treat the graphite/polycyanate (Gr/PC) composites as a new material. Mike Dean concluded his comments by stating that the program manager will make the final decision to meet the available dollars and schedule. LtCol Obal suggested we might learn some lessons from toy manufacturers, who utilize a team approach and typically have a short design-to-product cycle.

Dr. Aswani provided a case study to illustrate his points: a one-for-one substitution of PAN-based carbon phenolic for rayon-based phenolic in a rocket nozzle component (p. C-8); material qualification and schedule were critical factors. Two approaches were eventually utilized. Approach A (p. C-9) first involved thermal analysis and some material feasibility and screening tests after which three candidates were selected and procured. No problems were noted up to this point. Difficulties arose when design properties, screening tests, and full-scale fabrication were occurring in parallel. These included the following (p. C-10): extensive porosity and delaminations in the full-scale components due to lack of process control; structural failure due to differences in full-scale properties and coupon properties; component redesign due to the now reduced thermal and structural margins; need for new nondestructive evaluation (NDE) standards. Several full-scale components had to be scrapped as a result of material deficiencies. This effort was discontinued after an expense of several million dollars.

Approach B, an on-going program, is following a more logical, step-wise procedure beginning with evaluating and selecting candidate materials and testing small subscale components built using those materials. After three candidate materials are selected, fabrication and processing methods are studied. When test articles have been fabricated and tested, a process sensitivity study is performed. Finally, subscale tests and full-scale tests are to be completed. By using this approach, "all the i's are dotted and t's are crossed before going to the next step." This approach also involves a nice balance between analyses and tests.

Mohan Aswani concluded his presentation by discussing the important factors for successful application of advanced materials. What constitutes an adequate material property database is somewhat dependent on the application. Relevant experience with similar materials and applications will also contribute to a successful application. Other

factors include a system engineering approach with a balanced allocation of requirements; realistic material trade studies; acceptable design and analysis practices; a well-planned test program with results correlated to analyses; and proven processing specifications and demonstrated producibility. Analyses and correlations with test data are the items most usually sacrificed. LtCol Obal remarked that fabricator experiences with particular materials need to be well-documented since people are always changing; otherwise the same mistakes will be made. Dr. Aswani pointed out the lack of communication between material suppliers, fabricators, and designers. One attendee commented that if companies were afraid of failure in development programs they should shut down; in these programs failure should be expected from which lessons can be learned. Bob Acree (Phillips Laboratory) asked how program managers could be convinced of acceptable factors of safety for composites (or other advanced materials). Dr. Aswani emphasized that safety factors should not be confused with material allowables. Safety factors, associated with design criteria, are established based on several conditions, i.e., test or no-test options, one vs. many systems, experimental vs. operational systems.

Though coupon testing is necessary, other tests such as of structural components and joints are also required. Laminate properties are typically derived from measured unidirectional properties then compared to test data. Dr. John Stubstad (BMDO) stated that an adequate materials database is coupled to a well-planned test program. For example, aluminum alloys are not tested much due to the large, available database and experience base; this is not so for composites. Everyone agreed that convincing a program manager is difficult. The Hughes approach is to understand the fiber well and develop an increased understanding of the polycyanate matrix. Ms. Burns reiterated her earlier comment that the experience/knowledge of Gr/Ep systems should be utilized so that any test plan for the Gr/PCs could be expected to be relatively simple. Another attendee commented that advanced materials are only used when absolutely necessary because it is so difficult to get them inserted. According to Pam Burns, Hughes' DoD customers do not want composites on their spacecraft, the primary reason being cost. For composite parts, proof testing is required. A need for a method to convince program managers that proof testing is not necessary was suggested. Dr. Louis Rosales (TRW) said that a program manager needed a reason for using advanced composites; their biggest concern is usually the payload. Dr. Stubstad believed that risk was a major issue: "Why take the risk when you don't

Producibility does not necessarily mean a quantity of thousands, just that two or three can be made the same.

have to?" Aluminum is a low-risk material because a large database exists and all the contractors have extensive processing/fabrication/assembly experience with it. Dr. Aswani commented in post-workshop notes that the reasons for using composites are increased performance and reduced weight. These two benefits should be achievable at "reasonable" cost and acceptable schedule risk.

#### B. DR. LOUIS ROSALES, TRW

Dr. Rosales presented TRW's perspective on advanced materials insertion into spacecraft. Important design requirements at TRW (p. C-14) include sensor instrument performance, dimensional stability, thermal management, and weight. Material properties that help meet these requirements are low molecular/moisture outgassing; low thermal expansion, high modulus, low matrix microcracking, high thermal conductivity, and high specific strength and stiffness. Advanced composites fulfill many of the material property goals. There is particular interest in high thermal conductivity fibers.

TRW is developing a wide range of sophisticated spacecraft busses with sizes ranging from <1000 pounds to >10,000 pounds (pp. C-15 - C-16). The design-to-product concept being used for these spacecraft involves participation of all relevant groups (design, materials, manufacturing) from the beginning. For reasons of standardization, basic building blocks or modules that can be tailored to specific applications are being developed. Advanced technologies are continually inserted in both the structure and payload. The first generation prototype has been fabricated from Gr/Ep; second generation spacecraft will be fabricated from PC-based composites.

Dr. Rosales also discussed requirements for ultra-lightweight, dimensionally stable components (p. C-17) such as antennas, metering trusses, and optical benches. Material selection is very important for these types of applications. They must be deployable and stable with respect to atmospheric moisture/vacuum dry-out, temperature changes, temperature cycling, and vibration or transient loads. Advanced composites have some utility for these types of components. Materials of interest include high stiffness graphite fibers (up to 130 Msi); matrix materials such as polycyanates, modified epoxies, and co-polymers that absorb little moisture and are microcrack resistant; and ultra-thin plies (<0.001 in/ply) for zero CTE laminates. However, a material database is necessary before new matrix materials and fibers will be used.

Joint tests are being done for every possible configuration. A full-scale bus has been built and tested. TRW cannot wait for B-allowable data to be developed for a

material. 5 LtCol Obal asked about the as-delivered quality of the Gr/PC prepreg material. Dr. Rosales replied that TRW is developing a procurement specification for Gr/PC prepreg. Since they are working closely with the vendors quality was pretty good although there was some fiber variability. LtCol Obal commented that the structure typically represents about 15 percent of the total spacecraft weight and it may be possible to save a few percent (3-4 percent) by using composites. He asked if spacecraft primes were interested in something that could be made quickly a la assembly line style, production quantity spacecraft. The response was that every spacecraft is different, at least at present. For high-tech spacecraft, system life is important: extra fuel can be stored or, perhaps, payload performance may be improved and made flyable for a longer period of time if the structure is lighter weight. Louis Rosales stated that weight was a major driver in all the programs for which TRW was competing. Dr. Aswani remarked that system level trades are typically not done: cost is evaluated by determining the purchase cost of the structure relative to aluminum. With the trend toward higher and higher modulus fibers, a question was asked regarding the benefit/cost of such improvements. According to Dr. Rosales, that is presently being worked out. Pam Burns indicated that for launch costs of \$30,000/lb, the structures people at Hughes were excited about the possibility of saving 20 lb on a 3,000-lb satellite. The step function is still the launch vehicle: if weight goals cannot be met, a larger, more expensive launch vehicle is necessary.<sup>6</sup> Mr. Harry Dursch (Boeing) also said that weight was not the only driver. LtCol Obal then asked if there was some issue/technical difficulty that prevented TRW from throwing away Gr/Ep composites and replacing them with Gr/PC to which Dr. Rosales responded that Mr. Rich Lewis (TRW) would address that subject later.

Dr. Rosales then presented some data comparing properties of PC and Ep resins (p. C-19). Dr. Aswani asked if there was a system impact for reduced outgassing, a property which is usually highlighted as an advantage for the PC resins. Though it typically means less degradation and longer life for the specific element, the system impact was probably minimal. For dimensionally stable structures moisture absorption (p. C-20) and microcracking (p. C-21) are more critical. Ms. Burns indicated that Hughes found Gr/Ep unsuitable for structures in which dimensional stability was critical although Gr/TPs, for

<sup>&</sup>lt;sup>5</sup> "A-allowable" refers to a mechanical property value above which ≥99 percent of the data will fall with a confidence of 95 percent. "B-allowable" refers to a mechanical property value above which ≥90 percent of the data will fall with a 95 percent confidence level.

When weight is not an important factor cost usually is.

which the Gr/PCs are replacements, are suitable. Mike Dean mentioned coanngs, often used to protect polymer composites in space, which usually result in additional design costs. Dr. Rosales concluded that more testing was needed, particularly for thermal cycling and microcracking. In terms of manufacturability (p. C-22), the PCs are comparable to the epoxies with better "out-time" but the database is still limited. A concern was raised about the environmental aspects since the material contains cyanates. According to Louis Rosales, the material is "environmentally sound" and can be handled as other Gr/Ep composites—it does not require special handling.

TRW has nearly 5 years of experience with the Gr/PC materials, mostly in component fabrication and test rather than material development. Tubes, membranes, and honeycomb composite parts have been made. Significant amounts of processing data are being generated during fabrication of subelements for their advanced bus initiative. A design database, necessary to design for weight efficiency and to determine safety margins, is being developed in parallel with the AXAF (Advanced X-ray Astronomical Facility) contract. TRW requires sampling from three lots for B-allowables. Both PAN- and pitchbased fibers, in tape and cloth forms and with a wide range of properties, are being evaluated in conjunction with the raw material/prepreg suppliers. TRW also "spot checks" data generated by other companies as part of a data-sharing effort. It was suggested that the government might be able to help coordinate such data-sharing efforts though not necessarily providing funds for such efforts. Mike Obal thought this "may be the wave of the future given the limited resources." Another attendee commented that the United States needs to do this for competitive reasons. Designers need access to the data a la the Military Handbooks. Someone asked what it would take for designers to use a databook. The response was that standardized test procedures were needed as well as an independent source to evaluate the data in such a book. Pam Burns said that Hughes works with the material suppliers to generate data. Martin Marietta (MM) generates allowables for unidirectional and 0°/90° laminates. A verification test of the final lay-up is also performed. It was then stated that the government pays for 90 percent of the data anyway since most of it is generated on Independent Research and Development (IRAD) programs. The issue of use of Japanese or other foreign graphite fiber was raised by Dr. Mike Rigdon (IDA). Use of PAN-based fibers is already controlled, although it is possible to obtain a waiver, such

<sup>&</sup>quot;Out-time" is the amount of time a prepreg is exposed to ambient temperature (outside the freezer). Increasing out-time usually results in decreased drape and tack of the prepreg while allowing moisture absorption. "Tack" is the level of stickiness of the prepreg. The ability to maintain the appropriate level of tack for lay-up is generally affected by time, temperature, and humidity conditions.

requests are handled on a program-by-program basis. Pitch-based fibers will also be controlled, the object being to protect Amoco after the patent expires. Dr. John Tracy indicated that McDonnell Douglas was told by NASA that Japanese fiber will not be used/allowed. It was thought that such restrictions would limit the selection to relatively low modulus fibers but Mr. Dave Powell mentioned Imperial Chemical Industries' (ICI) 65 Msi PAN fiber-reinforced prepreg.

#### C. DR. TOM DRAGONE, ORBITAL SCIENCES CORPORATION

Dr. Dragone presented the small satellite designer's perspective. Orbital Sciences Corporation (OSC) develops and builds advanced design concepts for lighter, faster, cheaper launch vehicles a la Pegasus. OSC's design philosophy is to utilize state-of-the-art technology whenever possible, "even if extensive heritage doesn't exist." Emphasis is placed on rapid design through development to production. This means early prototyping and extensive testing. In Dr. Dragone's words, "I don't care how many coupon tests I get. I want parts I can test, bend, etc." Since a major focus is reduced cost to orbit, off-theshelf materials are used as appropriate. However, if specific, well-defined performance advantages can be obtained using advanced materials they will be used. He stated that modulus and strength are the primary design drivers for Pegasus. Mike Obal commented that OSC risk was based on testing and wondered if prototype testing was more cost effective than other approaches. Dr. Aswani inquired how the materials performance was analyzed with respect to test data. Dr. Dragone replied that coupon test data are needed during the preliminary design and material selection phases of a program and that OSC's emphasis is more on test than analysis. Mike Dean remarked that when a part was built the process variables would affect structural performance and then asked how OSC determined that the part would be made the same way the next time. Tom Dragone replied that the prototype parts are fabricated by processes as close to those used for the production component as possible.

Dr. Dragone described several of the vehicles that OSC produces (p. C-28): Pegasus, PegaStar (i.e., SeaStar), and MicroLab Small Satellite Bus (i.e., ORBCOMM). The Pegasus (p. C-29) is an all-composite structure (motor case, wings, fins, and nozzles) containing advanced propulsion, avionics, and thrust/vector control (TVC) technologies. PegaStar, a 500- to 800-lb satellite, uses off-the-shelf materials (p. C-30). The MicroLab bus, 100 to 200 lb, uses some unspecified advanced materials. LtCol Obal asked what factors would be critical for material selection if a spacecraft were ordered tomorrow.

Dr. Dragone replied that availability would be essential due to the short turn-around time typical of their programs. Fabrication work would have to be subcontracted. Any components manufactured by the subcontractor would have to meet the schedule, property, and performance requirements. Qualification and acceptance testing would also be necessary. Mohan Aswani questioned OSC's methods for determining that a subcontractor can indeed use the selected material, to which Dr. Dragone responded that the subcontractor would have to show that he has experience with and control of the process.

Dr. Dragone described OSC's design drivers (p. C-31) for launch vehicles (short life cycles and uniformity/manufacturing concerns) and satellites (long life cycles and unique mission constraints). The importance of mechanical and physical properties<sup>8</sup> (p. C-32), hygro-thermal properties (p. C-33), environmental properties (p. C-34), and other considerations (p. C-35) is indicated for each case, though specific values are not provided. A video showing a Pegasus flight illustrated the harsh environment to which advanced materials are exposed on launch vehicles. LtCol Obal asked if the dark material in the area of the shroud was a contaminant to which Dr. Dragone replied it was probably debris from the explosion during shroud separation. Mike Dean remarked that contamination from material outgassing during the short-term launch environment was minimal compared to that in the actual on-orbit environment. McDonnell Douglas's experience shows that separation systems are not contaminating although outgassing is a problem; cleaning the fairing (for the Shuttle) is critical. Another attendee indicated that offgassing of adsorbed species and particulate contamination are more of an issue than outgassing of absorbed species. To conclude, Dr. Dragone mentioned that cost, availability, manufacturability, safety/health concerns, and handling concerns were key factors affecting design. Interestingly enough, heritage is considered more important for satellite design than for launch vehicle design.

## D. MR. MIKE DEAN, BALL AEROSPACE

Mr. Dean discussed an on-going advanced spacecraft design project at Ball Aerospace. He noted that small satellites (i.e., compatible with a Pegasus) push the use of composites because of the weight driver: the weight fraction of the structure has to be as low as possible,  $\leq 5$  percent. Mike Dean commented on the need for functional duplication

LtCol Obal asked about the tailorability of damping requirement. Dr. Dragone stated that OSC would like to have damping integral with the panel. SeaStar is a stiffness-driven satellite and its structural damping is low (<1 percent).

in the sense that it is desirable for the structure to serve more than one purpose. For example, it is desirable that a composite structure be capable of providing thermal control and electro-magnetic interference (EMI) shielding as well as supporting the necessary mechanical loads rather than having different materials for each function with a corresponding weight increase. Ms. Burns indicated, however, that cost of the composites was a significant issue. Mr. Dean remarked that the launch vehicle drives the up-front costs. Composite costs of \$200,000 to \$300,000 per spacecraft may save a \$20 to 30 million cost increment if a smaller launch vehicle can be used. LtCol Obal asked if spacecraft assembly is an issue for composites, to which Mike Dean replied that it was for satellites within the weight margins of a Pegasus.

At Ball, small satellites are thought to be necessary. A full mission capability—power, guidance, and other support subsystems—with high efficiency and performance is assumed. Cost-effective operation is also key although he remarked that program managers often react too quickly (and, often, negatively) based on 20–30-year-old materials data. Mr. Dean indicated that establishing flight heritage was important, especially to increase the program manager's comfort level and to reduce risk.

Cost is obviously an important concern for a program manager. Mr. Dean presented cost figures for Gr/Ep composites based on joint NASA/DoD data obtained from 10 years of experience on fixed-wing aircraft (p. C-39). Material cost is a small percentage of the total, 3 percent, while fabrication, assembly, and inspection represent much higher percentages of the total—27, 28, and 27 percent, respectively. To be low cost, then, means that 90 percent of the costs must be controlled, or in other words, the fabrication, assembly, and inspection costs must be controlled. One attendee pointed out that material selection and design, though a small fraction of the total, drive the fabrication, assembly, and inspection costs. Mike Obal inquired if the cost data for Gr/TP structures was similar, to which Mr. Dean replied it probably was. For the Gr/TPs, capital investment cost is a significant factor to achieving low costs; it is not included in this cost model. Dr. John Stubstad asked if the chart for Al structures would be similar and it was thought it probably would be. According to John Tracy, McDonnell Douglas has shown costs can be decreased via part count reduction that is achievable with composites. LtCol Obal then remarked that Al and composite designs needed to be compared using this cost breakout/

I believe this would only apply if satellite weight margins were borderline. If weight margins could be easily met with existing materials like Al there would be no advantage to using composites.

Tooling and design represent, respectively, 7 percent and 8 percent of the cost.

format. The difficulty in moving beyond the "design-and-fly-one-at-a-time" mode of thinking was highlighted as another factor preventing widespread use of composites. The spacecraft mission is an important cost driver since it essentially determines the launch vehicle. The point was reiterated that the launch vehicle is the primary determinant affecting satellite weight/cost in order to keep below the step function increase in cost caused by a larger launch vehicle. Additional experience and increased communication among designers, material suppliers, and fabricators will allay some of the concerns with advanced composites. Mike Dean mentioned that fastener costs are high, anywhere from \$200 to \$300 per installed fastener. Bonded assemblies such as would be found in a composite satellite may lead to reduced costs and weight. A multibuild assumption remains "a big bugaboo" since composite structures become cost competitive with metal only beyond 3 to 5 units; it is not clear that such a goal will ever be reached for space programs.

Material requirements were discussed in terms of mechanical properties, system design, fabrication, and assembly (p. C-40). Strength allowables for minimum weight are determined by first-ply failure or residual strength at ultimate failure. Random launch loads make it difficult to predict the primary loading direction. Compression properties are driven by the matrix. Compression strength is not typically a design driver but modulus is. Other properties of interest are related to the mission orbit environment and payload parameters. At this point LtCol Obal asked if there was any intent to get away from protective coatings for composites exposed to the space environment. Mr. Dean replied that uncoated composite facesheets were being examined for use as radiators. This radiator study is part of the integrated system design effort which appears to be related to multifunctional structures. Electro-magnetic interference (EMI) shielding is to be considered next. The projected lifetime on orbit will also affect any decisions to use coatings for protection: for a 20-year lifetime, coating may be necessary while for a 1-year lifetime it may not be. In terms of manufacturing, being able to predict costs based on one-at-a-time production levels is expected for the immediate future. Mike Dean thought that Gr/TS processing experience was sufficient to aid in cost predictions. He also indicated that intelligent processing methods were not necessary.<sup>11</sup> The 5- to 10-year evolution in satellite design is common. It is very expensive to scrap parts when designs change late in

It is not readily apparent that this is true. For large production runs intelligent processing may not make sense but for small production runs, as is currently the case for spacecraft fabrication, and/or flexible manufacturing lines (meaning a number of different materials are fabricated into parts on the same line) it probably does make sense. It is absolutely critical to understand the material and process to ensure that the same thing (shape, properties, etc.) is made from one run to the next whether several hundred parts or a single part are made.

the system development process, so it is important to make allowances for that. Mike Obal then commented that designs appeared to be limited to particular configurations. Mike Dean replied that these configurations were driven by assembly issues: for example, removable panels are needed to access the satellite interior. LtCol Obal then suggested that different shapes such as an innovative exoskeletal design could be examined. However, real estate is still needed to mount boxes, the general conclusion of the attendees being that industry has been conservative in looking at new designs. Dr. Bill Harvey (Rockwell) stated that thermal loads are next in importance to structural loads. Flat surfaces are necessary for providing paths to conduct heat away from the mounted equipment boxes and racks.

Mr. Dean presented some IRAD data on EMI shielding for several graphite fiber-reinforced composites<sup>12</sup> as a function of frequency (not included) and compared it to data for a 0.25-inch thick Al baseline structure.<sup>13</sup> Radiation shielding as a function of atomic mass was also briefly discussed. Other design factors needed consideration, too. It is clear that mission operational requirements must be well-defined up front so that potential problems can be addressed as early as possible. Mike Dean indicated that AC shielding is probably handleable, while the DC shielding is much more difficult. Mike Obal asked if foils were being embedded to handle EMI. The response was that it was difficult to do with frequent design changes and one-at-a-time fabrication methods. Fillers and additives to provide electrical continuity for adhesives were mentioned as an issue that needed to be addressed for polycyanates.

Design versatility of the composites was illustrated with a "star chart" on which each leg represents a particular property (p. C-41); the axes for each leg are normalized to Al. The area enclosed by the thick black line is 6061; the shaded area represents Gr/Ep composites. Values for EMI and electro-static discharge (ESD) properties are, respectively, 3 to 10 times less than those for Al. Further development is needed to

<sup>12</sup> Different graphite fibers were used in each composite.

A recent article discussed the mixing of hollow conductive microspheres with a polymer matrix to create an electrically conductive syntactic foam for EMI shielding. Uncoated and Ag-coated SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> microballoons (10-75 μm diameter) and uncoated carbon microballoons (5-150 μm diameter) were mixed into an epoxy matrix. The volume fraction of particulate was about 70 percent. Some EMI tests were done and gave reasonably good results. But there are a number of practical issues to be addressed including mechanical properties and integration with fiber-reinforced composites. Reference: D.W. Radford and B.C. Cheng, "Ultra-Lightweight Composite Materials for EMI Shielding," SAMPE Quarterly, 24 (4), July 1993, pp. 54-61.

address radiation shielding and electrical ground concerns associated with composite use in space.

Reliability issues are of major concern to a program manager, particularly in terms of minimizing cost and schedule risks. This implies that the company has developed realistic options to meet the desired constraints and can, indeed, deliver. Risks can also be reduced via ground qualification testing, i.e., material characterization, component and sub-assembly functional tests, and system-level vibration and thermal/vacuum tests. Verification should be accomplished by a space flight. LtCol Obal indicated that this sort of approach is not viable given the current budget environment and thought that more credibility should be ascribed to ground tests. Mike Dean concluded his presentation by describing subject areas for further R&D. These include examining spacecraft design in light of emerging technologies/concepts such as "smart" structures, autonomous control and optimization for minimum weight. Nonstructural design standards need to be implemented for composites. These may include EMI/ESD behavior, empirical design relationships, and analytical codes for multicomponent, anisotropic material systems. Mr. Dean still believes that flight experiments are necessary for verification.

# E. MR. GORDON RITCHIE, LOCKHEED MISSILES AND SPACE COMPANY

Mr. Ritchie began with a comment on tooling for composite part fabrication, stating that tooling costs per satellite were expensive for most satellite programs but in the noise level for aircraft. LtCol Obal inquired what the cut-off point was for 40 to 50 satellites. Some work by TRW on Gr/TP composites showed that the break-even point (number of parts vs. cost), relative to Gr/Ep, depended on the specific part; for the parts TRW fabricated the number ranged from 10 to 50. Mr. Ritchie indicated that the IRIDIUM program, a multibuild satellite program, was not using Gr/PC composites as of early 1993.

The Theater High Altitude Area Defense (THAAD) program was, however, considering a Gr/PC shell to cover the entire length of the missile and, perhaps, for the bulkheads (p. C-46). A preliminary design, on p. C-47, lists some of the material and product form requirements in terms of ply orientations, number of plies, and thicknesses. Hundreds to thousands of these missiles are to be fabricated. Projected lifetimes on-station/in-storage are up to 20 years (before use) though the actual operating lifetime is only 5 minutes or so (p. C-48). Mike Obal asked if any metals were being used in the trade studies. Mr. Ritchie did not know. The temperature range over which system operation is

required reaches 450 to 550 °F. Moderate to high strength and stiffness are required, although relative to satellites the stiffness-to-strength ratio would be lower.

The number of satellites that might be built by Lockheed for a contract varies considerably, but is typically one or two (p. C-49). Storage lifetimes also vary considerably although operating lifetimes on orbit can be quite long, on the order of years. Moisture pick-up varies in different thicknesses of material. The temperatures of interest range from -250 to +250 °F. Mechanical property requirements include high stiffness and moderate strength as well as dimensional stability [coefficient of thermal expansion (CTE), coefficient of moisture expansion (CME), and microcracking due to thermal cycling]. Low outgassing would also be a concern. Mr. Ritchie pointed out that the specific properties of interest depend on the application.

A number of general concerns, relevant to a number of different applications, were identified (p. C-50). The lack of a material property design database was highlighted by several attendees. There seems to be a particular concern regarding matrix-dominated properties. The lack of a design database for joint properties—bonded or bolted—is another factor limiting acceptance of advanced composites. The issue of limited flight experience was also raised a couple times and generated some discussion. Mike Obal commented on the process of getting a Gr/PC satellite into space. Apparently no evidence of structural failure has been seen if there is adequate material, subcomponent testing, etc., prior to flight. Several attendees identified failures with composites: Concorde flight rudder; a composite fairing in which the face sheets blew off the core; and an unvented sandwich panel that exploded. Most composite designs to date have been very conservative in order to prevent failures. The lower tack of the PC composites is a manufacturing issue but is thought to be acceptable. John Tracy mentioned that McDonnell Douglas had experienced problems with low viscosity during the curing cycle. The fiber supply and international competition were also cited as issues. The M40J and M60J Japanese graphite fibers were highlighted as high performance fibers. The availability of domestically produced, advanced fibers comparable to M40J and M60J is limited due to the small markets. Domestic material suppliers have also been entering and leaving the market with apparent regularity, Amoco being one of the more recent in the prepreg business. And resin cost may be related to supply and demand. Pam Burns indicated that PC resin costs were about the same as those for epoxies with fiber being the high-cost prepreg component. One of the material suppliers stated that epoxy resin costs ranged from \$2 to \$40 per pound while PC resins ranged from \$13 to \$100 per pound, depending on quantity and sophistication. Fiber costs, particularly for the high modulus fibers, are considerably greater.

THAAD is concerned about the effect of post-cure processing on mechanical and physical properties (p. C-51). Their limited test matrix includes 5 coupons per test for 10 or 12 different tests, all performed at several temperatures. LtCol Obal remarked that looking at a subset of the entire set of possible test data was an appropriate approach. For spacecraft applications, concerns include the lack of data on dimensional stability and on contamination due to outgassing, and availability of thin ply prepreg (<0.002 inches) containing the ultra-high modulus fibers.

Gordon Ritchie identified particular materials of interest (p. C-52): THAAD expressed interest in the Hexcel F475 matrix with the M40J and M60J Japanese fibers. Given the recent legislation on foreign vs. domestic fibers it is not clear the Japanese-made fibers can be used. Other matrix materials include YLA RS-3, Fiberite 954-2A and 954-3, and the Amoco 1939-3 (which may not be available). In addition to the Japanese fibers, T300, P75, and P120 are also being considered.

Mr. Ritchie made it clear in his concluding statement that more data on the PC materials were needed. LtCol Obal suggested that the prime contractors should send him a list of specific data of interest. From his perspective it appeared that the assumption that no one else's data is correct is often made at prime contractor locations. He noted that this perspective may change in the future to reflect increased communication among competing groups along with reduced research funds.

#### F. MR. BOBBY HANSON, MARTIN MARIETTA

Bobby Hanson described Martin Marietta's perspective on advanced composites in spacecraft. He first presented a list of "nice" properties such as low outgassing and moisture absorption/strain and high toughness and glass transition temperature  $(T_g)$  (p. C-55). Of the listed properties only moisture strain and  $T_g$  are considered true design parameters. He then asked why Gr/PC composites would be used. One reason is that familiar processing methods a la Gr/Ep composites can be used. The other is that the cost impact is negligible: costs per pound for prepreg (three resins, same fiber) from the same vendor ranged from \$310 for Gr/PC to \$305 for Gr/Ep to \$295 for Gr/toughened epoxy.

The launch environment usually governs material selection and structural designs, important properties being stiffness, strength, and low cycle/high amplitude fatigue.

Mission performance and on-orbit operations may also affect materials and structural design. Integration, test, and handling concerns seldom drive material selection and design, although, since people are involved in the fabrication and assembly of the satellite, the material has to be tough.

Mr. Hanson then provided details on requirements for more specific applications: precision truss structures (p. C-57); nonprecision box structures (p. C-58); atmospheric entry forebodies (p. C-59); and thermal management components (p. C-60). Pictures of representative structures (not included in this report) were also shown. Only one example is highlighted here. Requirements for the precision truss include launch stiffness and strength, dimensional stability (near zero CTE, low hygroscopic strain, low microcracking, and creep resistance), and low outgassing. The strength, stiffness, and near zero CTE requirements drive fiber selection; the other requirements drive matrix selection. A precision truss is currently in flight. Mike Obal asked if any creep data had been generated to which Bobby replied no. LtCol Obal then inquired if the available data were adequate. Mr. Hanson answered that, although microcracking and outgassing data were needed, he was happy with the existing data because most of the identified "nice" features of Gr/PC composites were not design drivers. Pam Burns commented that program offices often ask what the disadvantages are so she wanted to know if something was being missed. LtCol Obal responded that he had not heard a showstopper yet. Dr. Aswani remarked that showstoppers will not be identified until a structure has been designed, built, and tested. Martin Marietta has measured thermal cycling performance on coupons, tubes, and subassemblies for 500 cycles using an M55J/PC composite.

Bobby Hanson discussed some of the issues related to qualification testing and material maturity. Approaches for qualification testing vary from company to company and from application to application. As is true for most materials, a more complete database for a given composite increases its chance of being used. He provided several examples: 5 tensile and 5 compressive lamina tests as well as 1 proof test were required for secondary structure in the Gamma Ray Spectrometer; on the other hand, an extensive database was generated in a classified program involving a large composite tube to hold an antenna. LtCol Obal asked what was meant by a complete database, the response being that it should be broad rather than deep. Some data such as EMI and on-orbit life are difficult to obtain without accelerated tests. Mike Dean indicated that accelerated testing is

A minimal cost impact is a particularly important factor for these types of structures. Mr. Hanson noted that some customers are standardizing around PC composites rather than Ep composites.

typically done for point designs. Some of the companies participating in the workshop were developing at least initial exposure data through joint programs. There is no substitute for Father Time, however. Flight heritage can provide some of that data. It is also important because it means someone has been the guinea pig and has been through the process of design, fabrication, assembly, qualification, etc. But any part that is flying may be considered a demonstration of technology. According to Mike Dean, though, a flight experiment proves that a part/structure acted predictably.

Material maturity issues are relevant to all advanced materials. Material availability and product consistency are continual problems. The regularity with which suppliers of new/advanced materials enter and leave the market is a major concern. Material supplied two years in a row may not actually be the same material; there may be slight improvements that change the processing conditions or some other property enough to invalidate all the generated data. The market problem is critical for the small material suppliers, particularly when working with big companies on long-term projects. Pam Burns remarked that the advanced composites market for spacecraft is probably always going to be small, maybe a few thousand pounds per year. The market for aircraft may be significantly larger in comparison, on the order of 10,000 lb per month. With a new material one is also concerned that all the issues may not have been identified. As is frequently observed, the first thing one hears about a material is usually the best thing one ever hears.

Bobby Hanson concluded by saying that the Gr/PC composites were not enabling (p. C-62). The fact that they may offer increased robustness and reliability, though, makes them more attractive to program managers.

#### G. MR. JOHN COONEY, SPACE SYSTEMS/LORAL

Mr. Cooney presented a completely different perspective, that of a commercial satellite manufacturer. He began by commenting that Space Systems/Loral (SS/L) has over 100,000 composite components in space. Current major satellite and space system programs include INTELSAT, GOES, N-STAR, SUPERBIRD, Space Station, and Globalstar (pp. C-64-C-65). The GOES system is currently being fabricated; parts to be made from advanced materials are illustrated on page C-66. Its flight is expected in early 1994. For the next generation INTELSAT system, increasing numbers of parts are being fabricated from composites (p. C-67); the structure of version VII (1993 flight) will be about 60 percent composites by weight (p. C-68). In the future it is expected that composites could represent about 90 percent of the spacecraft structural weight.

LtCol Obal asked what the drivers were for using composites. Mr. Cooney replied that weight and payload were the most significant. Government requirements for early satellite programs did not drive Loral to use composites. LtCol Obal inquired about design/qualification constraints with respect to insurance. Mr. Cooney indicated that problems for commercial companies are similar to those for companies supporting military needs. He did state that designers are more difficult to convince than program managers: flight heritage is, for designers, a "religion." Once a fiber/matrix combination that works is found, there is a tendency to stick with it.

SS/L has been working Gr/PC materials since 1988, initially under IRAD efforts. Material specifications for and fabrication of Gr/PC flight hardware occurred in 1991. And their first INTELSAT flight will be late 1993. The current composite material of choice is a Gr/934 epoxy (p. C-70). Since it performs adequately there has been reluctance to change. However, there are some issues which can be addressed by Gr/PC composites (pp. C-70-C-71). The desired requirement that there be no process changes is considered essential. Though the typical PC shown on page C-72 has a lower tensile modulus, it meets or exceeds the other requirements. SS/L has tested a large number of materials, in both tape and fabric form, from Amoco, ICI Fiberite, and YLA (p. C-73). A very specific set of mechanical properties tests (ASTM standards) have been defined for material qualification (p. C-74) including, among others, tensile strength, modulus, and Poisson's ratio; compression strength and modulus; environmental testing (mechanical properties) from -100 °C to +100 °C; void content; and laminate density. A set of physical property measurements are also required as part of material qualification (p. C-75): CTE, CME, outgassing, and resin-related processing features. Thermal cycling tests involve a minimum of one lot of material cycled 10X between -180 °C and +135 °C; 0° compression strength and modulus of the thermally cycled specimens are measured at room temperature. A summary of room temperature data<sup>15</sup> for a number of different materials can be found on page C-76. In geosynchronous orbits, radiation becomes a significant problem for composites. The only data Mr. Cooney could find on radiation effects was simulated data generated by Nippon using electron beam irradiation (p. C-77).

Fabricated hardware included a 16-foot foldable reflector, batter sleeves, radiator panels, solar array yoke, and hinges, among others (p. C-78). In response to a question from Harry Dursch, Mr. Cooney indicated the parts were co-cured rather than secondary-

With the exception of short-beam shear results, all the data have been normalized to a fiber volume of 60 percent.

bonded. While a few fabrication issues have arisen—lack of tack, <sup>16</sup> possible additional debulk operations—there does not seem to be an "Achilles heel" for Gr/PC, at least so far. And though SS/L is comfortable enough with the existing data and experience Mr. Cooney said it "would be nice to see the data" to back up this statement. As far as SS/L is aware there have been no failures of composite parts.

John Stubstad asked if DoD was more risk averse than the commercial sector. Mr. Cooney replied they were about the same although reliable performance for a long period of time was a particular concern of the commercial sector. Pam Burns explained that the reason for this is that DoD asks for more redundancy in its space systems, and performance envelope is pushed more for the payload than the structure. Mr. Cooney remarked that advanced materials are typically brought into a program at the beginning. While it is possible to bring changes into a program later it makes qualification more difficult. He also commented that it is easier to sell advanced technology on new programs such as N-Star; and, in fact, it is easier to do everything up front when a concurrent engineering approach is used. LtCol Obal commented that he sensed more enthusiasm for using advanced composites in the commercial sector than on the military side. Pam Burns commented that the commercial side is leading technology insertion although DoD requirements in terms of threat environment, altitudes, and precision performance may be more stringent. Dr. Stubstad then asked if the two sides, commercial and military, treated the government different internally. Typically the two groups do not communicate although that is apparently changing. Ms. Burns remarked that "the customer is always right" and that "electronics sells satellites." For a commercial company the ability to squeeze an extra month or more of operation time due to a lighter weight structure carrying more propellant means additional revenue; the military does not have that motivation. Dr. Rosales stated there was a bigger difference in management oversight between NASA and the military than between the commercial sector and the military. According to John Tracy, on some projects there has been one NASA engineer for every McDonnell Douglas engineer. Part of this is attributed to difficulties associated with man-rated space systems.

Mr. Cooney concluded his presentation by focusing on potential R&D topics (p. C-80). Included among these are effects of moisture and thermal cycling on dimensional stability, radiation and low Earth orbit (LEO) effects, and lower temperature curing systems with high T<sub>g</sub>s. He also believed that structural bonding adhesives needed to be evaluated.

<sup>16</sup> Mr. Cooney believes that with additional experience this may not be a problem.

And though he would "hate to see new material systems," it may be necessary to consider additives to the PCs for improving thermal and electrical performance. Mike Dean suggested that structural degradation over the mission lifetime and shielding have not been properly addressed. LtCol Obal asked if SS/L was interested in integrating electronics, etc., into spacecraft panels. Mr. Cooney replied that harness design represented a significant part of the overall spacecraft design so there might be some interest.

#### H. MR. BILL HARVEY, ROCKWELL INTERNATIONAL

Bill Harvey presented his manager's perspective of his perspective. He began by stating that designers avoid using advanced materials to meet the customer's performance requirements at the lowest cost and risk, and lowest cost for design, analysis, fabrication, and assembly, unless forced into it (p. C-83). In fact, Rockwell's sensorcraft baseline structural system is defined by aluminum. Using Al for the structure enables them to meet all of their requirements (p. C-84): manufacturing cost and schedule, mechanical and physical properties, weight, etc. Requirements for the sub-systems are also based on an Al structure. LtCol Obal remarked that he thought this was a dangerous approach: if, in a few months or more, the structure turns out to be too heavy, Rockwell will be in a bind since there was no parallel effort to examine advanced composites.<sup>17</sup> He then asked if there was a lack at Rockwell that prevented them from selecting another material. Mr. Harvey replied that his fabrication experience both inside and outside the company was one factor leading to the selection of aluminum for the structure; and his personal experience designing with advanced composites was another. Mr. Harvey's previous experience in the design, analysis, and fabrication of composite structures, i.e., for TRW's ACTS (Advanced Communications Satellite) program, enabled him to determine when "it is good value" to the customer to use composites and to select the appropriate materials for trade studies in order to design the most cost-effective structures. 18

In later comments Bill Harvey indicated that the Brilliant Eyes spacecraft will operate in a hostile, radiation-intensive environment. It was, therefore, designed to provide the necessary radiation shielding for the internally mounted electronics, etc. Since radiation shelding is a function of material density and thickness, a lightweight composite spacecraft would have a lower radiation shielding capability. And that offers no advantage to the customer, DoD. What Rockwell would be interested in are composites for structural subsystems such as solar arrays, optical benches, and orbital insertion, and, possibly, radiator or structural equipment support panels for batteries.

Bill Harvey later indicated that two quick trade studies have been performed using graphite-reinforced polymer composites for the BE structure. However, the only way these designs could be utilized is if a lightweight, spray-on, radiation-hard coating was developed.

Mr. Harvey did identify several unique applications where composites were necessary to meet the performance requirements (p. C-85). A SiC<sub>p</sub>/Al composite<sup>19</sup> was found to be optimum for facesheets and stiffeners in a sandwich panel, partially because "it's just like aluminum." Another application is for struts made from Amoco's P75/1939-3 tape; an orthotropic lay-up provides an optimum stiffness-to-weight ratio needed to meet the first mode frequency requirements. The third application is for a lightweight assembly of sandwich panels, designed to meet jitter, attitude control, and contamination requirements (pp. C-86 – C-87). Panels designed for twisting and bending are made from a Hexcel honeycomb core covered with [0°/±60°]<sub>s</sub> P75/1939 facesheets. Closeouts and local doublers for these panels are fabricated from T300/1939 lamina. Support beams are fabricated from an orthotropic lay-up of P75/1939, again to provet first mode frequency requirements. Rockwell has been successful in using the 1939 modified epoxy in terms of fiber/matrix interfacial bonding. The fact that it is available with the P75 fiber in thin plies is advantageous; and its low microcracking, outgassing, and water absorption are also attractive.

His request to the workshop audience was "tell us all the good stuff about why polycyanates are better." With that Mr. Harvey compared some TRW data on PC and Ep systems with the P75/1939-3 (pp. C-89 - C-90). As it turns out, the 1939-3 modified epoxy is actually a polycyanate so it is not surprising that there are few differences between it and the TRW PC. However, he asked if anyone had material qualification test data for P75/PC or P100/PC. One of the attendees wondered what was meant by materials qualification test. According to Bill Harvey, it means a statistical database and a process specification that allows those properties to be obtained so that reasonable predictions can be made regarding product performance. Another attendee commented that companies do sell data and process specifications to each other. Mike Obal thought that the data may already exist, and it was just a question of bringing it together in the right format. The reality is that knowing a material is space qualified, that it has been flown, is a significant factor affecting its future use. The idea of material qualification as a roadblock was reiterated in the last few charts (pp. C-92 – C-94). According to Bill Harvey, program managers do not have the funds and schedule time available to do material qualification for a particular program. Another attendee commented that it may be enough for an expert review board to determine if a material needs to be space qualified. If the committee decides yes, then the companies must figure out a way to do that as part of the program.

<sup>19</sup> Aluminum alloy 6090 contains 25 percent SiC by volume.

Dr. Aswani stated that developing process specifications for composites without actual experience was not a sound approach: "just because you know what it tastes like doesn't mean you can cook." When the experience base is lacking it is difficult to transfer technology from the laboratory to the designer. John Stubstad remarked that it traditionally takes 15 years for a material to become qualified. Bill Harvey then mentioned that P75/934 was qualified as part of TRW's NASA ACTS program (launched September 11, 1993); required data on another composite, P75/930, was provided over a 3- to 4-month time period. The question of what was meant by qualifying a material was still unclear. Mr. Harvey stated that data on strength, modulus (stiffness), distortions due to thermal and moisture expansion, outgassing, etc., were required before a material could be considered qualified. LtCol Obal indicated that if a data generation program were to be initiated all data collected would be sent to industry for comment. Members of the audience pointed out, though, that without the supporting information on processing, etc., the data are nebulous and difficult to interpret.

## I. MS. PAM BURNS, HUGHES

Hughes has been involved in the design and fabrication of a large number of commercial satellites, particularly for communications (p. C-97). Examples of upcoming systems include Astra 1C, Solidaridad (HS 601, 1993), and PanAmSat and SAJAC (HS 601, 1994). Design requirements (p. C-98) for these spacecraft include dimensional stability, high structural rigidity, minimum contamination, rapid tracking and pointing capabilities, deployability, survivability, immunity to electro-static discharge events, and low cost. The corresponding materials requirements were also described. For example, to meet dimensional stability requirements the material must have low CTE, high thermal conductivity, low hygroscopy, and minimal microcracking. One of the attendees mentioned the importance of thermal considerations in determining the material design. An organic composite containing graphite fiber with a modulus ≥50 Msi was stated to be acceptable for electro-static discharge requirements. Low cost requirements are addressed in terms of producibility, versatility in fabrication, efficient joining and fastening, and repairability of the materials.

Ms. Burns indicated that cost is becoming an increasingly important issue in maintaining U.S. competitiveness. Mohan Aswani inquired about the methods used to assess/measure costs since there are several possible approaches: system costs, aluminum vs. composite fabrication cost, or spacecraft bus acquisition cost. And how does one

convince a program manager that it is low cost? Ms. Burns indicated that there were three aspects: material cost, processing and integration costs, and system costs, all of which depend on the application. While material cost is typically considered to be small relative to fabrication, assembly, and inspection costs, a high material cost will eat the budget of that particular group. Pam did say that Hughes' experience shows that composite components can be more expensive than Al ones but the overall system cost may be lower if one utilizes the advantages and flexibility of composites in the design. John Tracy mentioned that McDonnell Douglas has data to show that composite component cost can be lower. The track record of material suppliers (those entering and leaving the business such as Amoco (prepreg) and DuPont (fiber)) was mentioned as a concern related to maintaining domestic sources for advanced composites.

In any case, Hughes is interested in the Gr/PC composites for cleaner spacecraft, reduced weight and geometric distortion, increased toughness, and improved radiation resistance (p. C-99). A number of applications were identified (p. C-100): stable structures such as trusses and antenna reflectors; lightweight, noncontaminating structures such as optical benches; Al and Be replacements; and missile structures. Hughes is attempting to implement the Gr/PC composites by taking advantage of their similarities to Gr/Ep composites. Some parts have been fabricated for flight (p. C-101), for example, a P100/PC tube<sup>20</sup> for flight on an AF satellite next year. No compatibility problems have been observed between the Gr/PC and Gr/Ep composites: the technicians cannot distinguish between the two. Concurrent engineering, accomplished by teaming with material suppliers and fabricators, is being utilized to implement composites on their new 3-axis stabilized satellite, and it seems to be an optimal approach.

Ms. Burns described the qualification tests in some detail (pp. C-102 – C-105). Testing is performed in three stages, the first stage being to generate the design database and to verify wettability and property translation. Two different lots of prepreg must be tested. Design allowables are defined as the measured property less 2 standard deviations; for B-allowables 16 coupons are typically tested. A number of mechanical and physical properties as well as adhesion properties, for which Hughes specifies ASTM tests, must be evaluated. System level testing is specific to the application and is performed on sub-element or full-scale prototypes. Such tests include bearing tests, flexural strength and modulus for honeycomb sandwich panels, thermal cycling, and dynamic loading. If the

This tube is a replacement for Be: a three-piece Be part was replaced with a one-piece part.

intended use is a critical application, full-scale assembled articles are also tested to failure. There seemed to be general agreement that if test results from a number of contractors could be pooled there would be higher confidence. Standardization of materials would go a long way toward increasing acceptance of advanced materials (material researchers and suppliers take heed). One way to accomplish this might be to treat military and commercial customers the same, a difficult task without some reforms in the way the government does business. The time and funds available for material qualification have decreased: in the past, \$100,000 for testing over a 2-year period was not uncommon; now, about \$25,000 for 1 year is more the norm. John Tracy stated McDonnell Douglas trades two standard deviations with the number of coupon tests a la Military Handbook 17: If the number of tested coupons is reduced the subtracted amount (-20) is increased, resulting in lower material allowables. Dr. Rich Lewis (TRW) commented on the high cost of attaching strain gauges to the composites. TRW uses 29 samples from three lots of material; if 16 coupons are used there is an additional knock-down factor. One can see from these limited discussions that there is no convention for material qualification testing among the different companies. Mike Rigdon asked if there was any interest in a Polymer Composites Information Analysis Center. The Structural Materials Selection Guide put together by Ketema under the M&S Program was identified by several of the attendees as one of the few sources of data on the most advanced composites.

Additional design information is still required before these materials can be used (p. C-106): attachment concepts for joining composites to other composites or to Al, particularly addressing machining and CTE mismatch; quality control and NDE; survivability in space; and repairability. Better quality control and NDE methods may lead to lower costs and increased user confidence.

Ms. Burns identified a number of technology gaps that currently exist for the Gr/PC materials (p. C-107). Included among these are long-term stability (creep), atomic oxygen erosion, cryogenic properties, safety requirements (handling during manufacturing), and other environmental effects. She concluded her presentation by stating that the major roadblocks preventing insertion of the Gr/PC materials were cost, a reliable and proven database, and technology transfer.

### 3. MATERIAL SUPPLIER PERSPECTIVES

Material suppliers were asked to identify information they needed from designers to insure adequate material development. They were also asked to provide current data on the graphite-reinforced polycyanate composites including material options (fiber architectures, matrix options) and availability/maturity (quantities produced), density, physical and mechanical properties, contamination and outgassing, microcracking and thermal cycling, natural space environment survivability (e.g., atomic oxygen, radiation). Manufacturability/producibility and joining issues, estimated current and projected costs, and other risks were to be discussed. Note that an extra section on Bryte Technologies prepreg materials has been added to this chapter to more completely cover the available PC prepreg systems.

## A. MS. TIA BENSON TOLLE, WRIGHT LABORATORY

Ms. Benson Tolle first discussed the domestic source pitch fiber issue. Public law 102-396, passed October 6, 1992, requires that a minimum of 75 percent coal and petroleum pitch carbon fiber for DoD needs must be procured from domestic sources by 1994. John Dignam (Army Research Laboratory) asked if this requirement would affect researchers directly or only the systems? Tia replied that it was unclear at the present time. The precedent for this is a public law passed in 1990 requiring that 50 percent of DoD PAN (poly-acrylo-nitrile) fiber purchases be from domestic sources (p. D-2). The corresponding DFARS (Defense Federal Acquisition Regulation Supplement) amendment further specifies that the PAN precursors be domestically produced (United States or Canada). It is possible for a contracting organization to waive the requirement with justification, e.g., a domestic source cannot meet the schedule, or the company has no/little experience. Or a company may request a waiver by identifying the circumstances, although a plan to qualify domestic material must be included. A DFARS amendment for pitch carbon fiber was proposed in April 1993 and has since been accepted. It states that for a deliverable product containing pitch fibers, 75 percent of the total pitch fiber content shall be domestically produced. As for the PAN fibers, the requirement can be waived by the contracting organization (with approval of the Head of the Contracting Activity) if the delivery schedule cannot be met. One attendee asked if the waivers were difficult to obtain.

Tia replied that she did not know. Another attendee commented that it was the "worst kind of protectionism" and would restrict designs. Representatives from TRW and McDonnell Douglas indicated they have experience in trying to obtain waivers. Another attendee commented that the Japanese preclude their fibers from being used on military systems although John Dignam remarked that it was possible to get around this. David Powell (ICI) said that the Japanese "seemed soft" on that point. A general discussion of high modulus fibers ensued. In the Title III program, P100 fibers having improved handleability are now being manufactured; these fibers have a minimum tensile strength of 450 ksi. John Dignam stated that tensile strength was inadequate for interceptors—600 ksi is desirable. Gordon Ritchie asked if any improvements were expected for the P120 or K1100 fibers. Ms. Benson Tolle replied that to her knowledge no changes were planned. Bobby Hansen commented that the compression strength for the improved P100 fiber was the same as that of the older material.

Ms. Benson Tolle was asked to provide an update on the Dow cyanate resin problem. Dow has been working the cyanate chemistry for nearly 15 years. The same pilot plant facilities have been used since the mid 1980's: the phenolic precursor is made in Dow's Freeport, Texas, pilot plant; the cyanation reaction is completed at Lonza's Visp, Switzerland, pilot plant. Problems in the cyanation process were experienced in December 1992. Meetings held in May did not resolve the issue but eliminated probable causes. It was believed at the time that the phenolic precursor might be aging during storage. As of June 1993, a plan was developed to address the aging theory. If success was achieved, Lonza would be able to produce the specified polycyanate by mid-July (pp. D-6 – D-7). If unsuccessful, Dow would probably exit the polycyanate business because a long, joint R&D project was not considered economically justifiable. According to Dave Powell, Ciba Geigy claims it will produce the reaterials and it has a precursor source. However, it will not enter the market until Dow says it is leaving. In response to a question from LtCol Obal, one of the attendees stated it would take about 6 to 9 months for Ciba to produce a Dow-like material; additional time would be required to prove the equivalency of the new materials with the Dow-produced material. Requalification of the new material is, in fact, an issue. Dave Powell indicated ICI had enough Dow-based material to last through a short, unspecified time period. It was not known if Ciba had purchased the

Apparently Lonza experienced reactor problems last summer after which a new reactor was installed. There was, therefore, a long time gap between runs. In addition, the precursor was 3 years old; previous precursors were only 1.5 years old.

rights to use the Dow technology.<sup>2</sup> The decision to continue or not is expected to be based on economic factors. Dow was expected to take the hard line: unsuccessful production runs would mean withdrawal of the material from the market. Any market pull for satellite applications would probably be insufficient to sway them from that course.

Note: Since the mid-June workshop, the cyanate resin has been successfully produced in two laboratory runs by Lonza. Pilot plant production was scheduled to begin in mid-July, with delivery at the Freeport, Texas, plant expected by early August.

## B. MR. BRIAN GILMORE, HEXCEL

Mr. Gilmore began his presentation by re-stating the well-known advantages of the cyanates: excellent electrical properties, good mechanical strengths, low moisture absorption, easy processability, linear CTE, and easy resin modification. Drawbacks include resin cost, base monomer availability, lack of extensive data, and other yet-to-bediscovered issues. In response to the stated workshop goals he discussed the information Hexcel needed from the designers to ensure the developed material meets their specifications (p. D-10). As one might expect, mechanical property requirements were high on the list. With respect to the fiber, knowledge of designer preferences for pitch or PAN fiber to meet the stiffness and thermal management requirements for the application was desired. Information on the form of the fiber, e.g., unidirectional tape or bidirectional fabric,<sup>3</sup> and thickness per ply requirements is needed. The structural design approach (laminate or sandwich) and the associated joining techniques (co-curing, adhesive bonding, fasteners) will also affect the choice of materials. Pertinent environmental effects are application dependent and would include the severity of thermal cycling, radiation exposure, and atomic oxygen exposure as well as the minimum allowable outgassing. Any processing limitations that the end user might have, i.e., temperature or pressure limitations for autoclaves, also need to be identified. And, if possible, cost sensitivities should be made known.

Mr. Gilmore then described Hexcel's polycyanate products: HX1566, HX1584-4, and F475 (pp. D-11 – D-15):<sup>4</sup>

The Ciba materials are chemically different from the Dow materials. The Ciba process allows the material to crystallize. Bryte Technologies utilizes the Ciba resins in their prepregs.

Hexcel is selling a graphite-reinforced honeycomb to Boeing for a thermal management application on the 777 aircraft: T300 fiber is used in one direction, P120 fiber in the other.

<sup>4</sup> The HX materials are in development.

- 1. HX1566 was developed for use on the Jet Propulsion Laboratory Precision Segmented reflector program. A database was generated using this resin with the ultra-high modulus Hercules fiber. Raw material availability was a problem at the time of the conference: the resin is based on the Dow chemistry.
- 2. HX1584-4 was developed for radome applications. Most of the data on this resin system was generated using quartz and Spectra fiber fabrics. It can be cured at 250 °F or 350 °F. This material is available and will become a standard product late in FY93. Screening tests of this resin combined with high modulus graphite fibers are being initiated. Gordon Ritchie asked if a post-cure improves the properties, to which Mr. Gilmore replied that it did.
- 3. F475, an epoxy-modified/toughened cyanate resin, was developed for space applications. It has been a standard product since 1992 and is currently being qualified for unnamed space applications. Therefore, a fairly extensive database is being generated. It is being evaluated by the THAAD<sup>5</sup> and EOS (Earth Observing System) programs and is to be tested aboard some Shuttle experiments.

Fibers are available in unidirectional tape or bidirectional fabric form (p. D-16). Hexcel has limited experience with thin tapes although thick tapes present no problems. Their current capabilities range from 70 to 300 gm/m<sup>2</sup> with tape widths ranging from 1 to 12 inches. Fibers available in tape form include Amoco's P75 and P120 and Toray's M40J and M60J. Handleability of the fiber affects its ability to be woven into fabric: thin fabrics may be a difficult problem. Generally, if the fiber can be woven a cyanate prepreg fabric can be manufactured. Current capabilities for fabrics range from 100 to 400 gm/m<sup>2</sup> with P120, M40J, and standard modulus fibers. LtCol Obal inquired about the methods by which information about potential markets is obtained. Mr. Gilmore replied that the marketing/sales people know what the users want.

Mr. Gilmore supplied data on HX1584-4 and F475 neat resins including glass transition temperatures (T<sub>g</sub>), moisture absorption, fracture toughness,<sup>6</sup> and density (p. D-17); outgassing (p. D-18); heating and cooling CTEs (p. D-19-D-20); water

A Ciba cyanate is also being evaluated by the THAAD program.

Fracture toughness of the HX1584-4 is a function of the cure cycle: 4 hours at 250 °F or 2 hr at 350 °F. Toughness data for both resins are based on 8 coupon tests.

absorption<sup>7</sup> (p. D-21); and dynamic viscosity analysis<sup>8</sup> (pp. D-23 ~ D-24). Prepreg properties were described somewhat qualitatively (p. D-17) in terms of tack,<sup>9</sup> out-time, flow, and gel time at temperature.

Typical properties of finished composites were also described. Water absorption of a 60 volume percent T800/F475 laminate (p. D-22) saturates at about 0.8 percent. Dry (p. D-25) and wet (p. D-26) glass transition temperatures were measured for IM7/F475 laminates via dynamic mechanical analysis. Mechanical properties for T650-35/F475 included open hole tension and compression, flexure, short beam shear (p. D-27). These properties were evaluated under dry room temperature (RT) conditions and some were tested under 250 °F wet and 350 °F dry conditions. Warp tensile and compressive strength and modulus properties were presented for M40J/F475 composites (p. D-28). Data for M60J/F475 composites included 0° and 90° tension and compression strengths and moduli, 0° short beam shear, and in-plane shear strength and modulus (p. D-29). Chemical stability of IM7/F475 laminates in isopropanol, MEK, trichloroethane, JP 4, and Skydrol has been evaluated as well. At RT no obvious effects have been noted, as evidenced by the strength data on page D-30. Capt. Bill Cameron (Wright Laboratory) asked if any testing had been done in hydrazine to which Mr. Gilmore replied no. Mr. Gilmore indicated that additional data were available on all three materials (p. D-31):

- 1. HX1566—thermal cycling, radiation exposure, microcracking, water absorption, and mechanical properties for composites (JPL report); and electrical performance properties of the neat resin.
- 2. HX1584-4—electrical performance properties for neat resin and for quartzand Spectra- reinforced composites; mechanical properties for quartz- and Spectra-reinforced composites; and CTE for quartz-reinforced composites.
- 3. F475—electrical performance properties of the neat resin; and mechanical properties of IM7-reinforced composites; and smoke density and gas toxicity.

Mr. Gilmore commented on resin and prepreg costs. Both are expected to decrease with increasing volume, though perhaps not in the next 2 to 3 years. The cyanate ester resins range in price from \$15 to \$100 per pound, with \$30 to \$60 being more typical of

After 90 days the resin saturates (-1.3 percent).

The HX1584-4 exhibits low viscosity at the peak temperature near 120 °C. F475 flow is more controllable than that of the HX1584-4.

Apparently problems with tack have been noted for the Dow resins.

<sup>10</sup> The IM7/F475 material is apparently attractive for radomes and for the European fighter as well as spacecraft.

the prepreg resins. Blending epoxies or thermoplastics with the cyanates may also reduce their cost. Prepreg cost is driven more by the cost of the high performance fibers. Longer production runs, term contract pricing, and reduced testing requirements may contribute to reducing prepreg costs. Test specifications currently "include everything but the kitchen sink." It is clear that a sensible subset of those tests needs to be selected.

Mr. Gilmore concluded his presentation by describing Hexcel's space-related activities (p. D-33). One of their materials, P75/F584, was flown on the passive LDCE (Limited Duration Candidate Materials Exposure) experiment on STS-46. Other experiments involving active monitoring are expected in the future. He reiterated that the Hexcel cyanate composites were being evaluated for EOS and THAAD. In addition, the IRIDIUM satellite constellation program will use Hexcel composites (F584 epoxy matrix).

## C. MR. DAVID POWELL, ICI FIBERITE

ICI has facilities located all over the world. Its space materials business is located in Tempe, Arizona, and represents a small part of a much larger business (p. D-37). This facility provides small scale, flexible production capabilities as well as technical expertise in the areas of chemistry, processing, and testing. Material prices are based on value and technical content/support to satisfy the customer's total needs. Material availability depends, to a great extent, on the existence of a profitable market segment.

Mr. Powell showed representative chemical bond structures for dicyanates (p. D-38): in the cured state the structure is an aromatic ring in which the OCN structures are the cyanates. The absence of hydrogen bonding results in low moisture absorption (p. D-39). Glass transition temperatures and flame retardancy are enhanced by the aromatic content. The low cross-link density results in high toughness and tensile elongations ranging from 2.5 to 6 percent for the base polymers. This large free volume structure also allows moisture to be quickly absorbed and desorbed.

All of the ICI cyanate materials have 954 prefixes (p. D-40): 954-2A contains unique thermoplastics (for toughening and flow control) in a co-continuous morphology; 954-3's are all-cyanate precursors (one of which is toughened) and exhibit the lowest hygrostrain. Minimum viscosities are >1500 cps<sup>11</sup> and about 100 cps,<sup>12</sup> respectively.

<sup>11</sup> Tape thickness varies with resin viscosity. Thin 954-2A prepreg tapes (<2.5 mils) require higher resin contents.

<sup>12</sup> Gravity and flexible caul flow problems are possible with the 954-3.

Product data sheets were provided for both materials (pp. D-41 – D-42). Neat resin properties are comparable although the 954-2A has a slightly higher tensile strength and better toughness. In order to offer the most opportunities for designers, ICI can supply prepreg with any fibers on the market today (pp. D-43 – D-44). Fiberite has extensive experience with the high modulus fibers (modulus values greater than 70 Msi), preferred for spacecraft applications. The 954 prepreg forms (p. D-46) include fabric up to 34 inches wide (p. D-48) and tape up to 24 inches wide (p. D-47) as well as towpreg for filament winding and tow placement. Mr. Powell noted a 10X increase in the market for 954 materials over the past year or so. A large number of lots of 954-2/-2A and 954-3 prepreg have been produced: 80 (>3,500 lb) and 90 (>6,500 lb) lots, respectively. These materials are being used in several major programs including commercial satellites and AXAF-I.

Mr. Powell presented some typical data on these materials including the following: neat resin moisture uptake at 160 °F (p. D-49)<sup>13</sup> and RT (p. D-50), <sup>14</sup> dimensional stability (p. D-51), and dielectric properties (pp. D-52 – D-53).<sup>15</sup> LtCol Obal remarked that moisture uptake is particularly important for terrestrial stability, low moisture absorption being desirable. Data were also provided on space environment-related effects: outgassing and contamination (p. D-54); <sup>16</sup> thermal cycling and microcracking (p. D-55); and oxygen plasma stability (p. D-56). The amount of microcracking is a function of the laminate architecture and of resin resistance to microcracking. According to Mr. Powell it appears to be a handleable problem for the thin plies. Terrestrial environment-related data include hot/wet performance (p. D-57) and solvent sensitivity (p. D-58). In response to a question from Mike Dean, the speaker indicated there were some data on the cyanate materials at temperatures below RT.

Mr. Powell discussed what ICI calls the "data iceberg" in which mechanical properties (effects) are affected by chemistry, prepreg processing, and materials science (causes). The speaker remarked that ICI tries to address the basic understanding of the materials, as illustrated on page D-60. Variables for resins, fibers, and processing are all examined to create the material science database.

Both neat resin materials appear to saturate at about 1.8 percent under the specified conditions.

<sup>14</sup> This graph shows that the 954-3 cyanate-based material exhibits much lower moisture absorption than the 934 epoxy.

Dielectric constants for 954-2A and 954-3 do not seem to be affected much by temperature or wetness. The loss tangent, however, does seem to be affected under 325 °F, wet conditions.

Total mass loss and collected volatile condensable material levels seem to be well within the acceptable NASA range for 954-3.

The thermoplastic-toughened 954-2A material was described (pp. D-61 – D-77). A very fine, co-continuous morphology seems to be the most efficient way to toughen the material without degrading compression strength. The morphology is quite stable as well. It is less susceptible to creep and, since the end groups are all reacted, it exhibits excellent solvent resistance. Process parameters for making 954 prepreg from any one of a number of fibers include line speed, temperature, and pressure (p. D-62). Flow, tack, drape, and gel time are among the prepreg features that can be measured. When asked by LtCol Obal if the toughened 954-2A would replace the 100 percent cyanate-based materials, Mr. Powell replied that it would depend on the specific application.

Data exist for 954 composites containing PAN, pitch, glass, and quartz fibers, though some of it is proprietary (p. D-63). A typical set of material characterization tests includes tension (p. D-64), compression and shear (p. D-65), and flexure, fracture toughness, edge delamination, thermal cycling and microcracking, and solvent resistance (p. D-66). As an example, tension tests for strength, modulus, and strain are done at RT for 0° and 90° specimens. Open hole specimens and a 25°/50°/25° lay-up are also tested at RT. In addition, tensile strength and modulus are evaluated under 180 to 300 °F wet conditions. Typical laminate data for pitch (pp. D-67, D-69) and PAN (p. D-68) fibers were presented. 17 Someone commented that there was a story going around that the cyanate-based materials were difficult to bond. According to Mr. Powell, it has not been observed in ICI's experience: good bond strengths have been obtained using a Hysol EA 9394 adhesive (p. D-70). He stated that any on-the-shelf adhesive could be used. 18 Jim Koury asked if ICI had any data on IM6- and IM7-reinforced cyanate composites. Mr. Powell indicated there was a large database. Apparently, the Phillips Lab tried to obtain prepreg with those fibers but the 4- to 6-week schedule on which they were operating could not be met.

The fact that 954 systems process like epoxies makes them especially attractive. Neat resin, compression-after-impact and open-hole-compression tests, and glass transition temperature are evaluated for cure flexibility (p. D-72). For example, cured neat resin features that are evaluated include  $T_g$ , water absorption, compression strength, flexure strength, modulus,  $G_{1C}$ ,  $K_{1C}$ , density, morphology, and cure and post-cure time and temperature studies. Out-time (p. D-73) of neat resin (p. D-74) and prepreg (p. D-75) and

Note that the data are normalized to 60 percent fiber volume except for strain and short beam shear strength.

<sup>18</sup> ICI has not received any phone calls from users indicating there are bonding problems.

its effect on resin characteristics such as viscosity and flow during cure are also evaluated. Typical cure cycles for 954-2A and 954-3 are shown on pages D-76 and D-77, respectively: 954-2A has controllable flow; however, after about 20 minutes, the viscosity of the 954-3 becomes so low it confuses the machine.

Mr. Powell presented performance/cost data for several material systems. There is a cost delta associated with using the cyanate esters now, ranging from +2 percent for a P120-reinforced composite to +25 percent for a T300-reinforced composite. ICI can prepreg K1100 fibers with the cyanate resins but such prepregs are very expensive. Also, Amoco apparently requires some sort of written agreement before selling the K1100 fiber. Mike Dean inquired about lead times for K1100 prepreg. Mr. Powell thought 8 weeks would be required to obtain the fiber, followed by 2 to 8 additional weeks to make the prepreg. Fiber and resin supply are major drivers for prepreg manufacturing turnaround. The speaker did state that ICI had enough material in stock to be able "to nurse the market along" until the Dow resin supply question was resolved. Pam Burns asked if the Amoco 1999 resin was very different from the other systems that had been discussed. The response was that it is quite comparable to 954-3 and YLA's RS-3.

No significant "Achilles heel" has yet been identified for the cyanate-based composites. In terms of risk, supply stability in terms of raw materials and prepregger demise was felt to be most significant (p. D-79). These two factors affect program schedules, and typically result in material requalification, higher costs, and fewer design options.

Mr. Powell concluded his presentation by describing inputs needed for materials development (p. D-80). ICI is developing a resin specifically for space applications and is interested in input from everyone including fabricators and designers. For example, the following information is desired from fabricators: acceptable cure cycles, flow control, bleed schemes, resin content and fiber areal weight, prepreg out-time, tack and tack life, prepreg drape and radius conformance, tool release, post-cure residual stresses, bondability and repair, and environmental issues.

Resin viscosity during cure appears to increase with increasing out-time; the temperature for gelation shifts to slightly lower temperatures with increasing out-time. This is observed for both the neat resin and the prepreg.

The baseline matrix material is a 934 epoxy resin.

# D. MR. GARY PATZ, YLA, INC.

YLA is a small company (15 employees) that produces fiber-reinforced RS-3 prepreg. Capabilities include production of 12-inch wide unidirectional tapes and impregnation of fabrics up to 54 inches wide via a hot melt process (p. D-82). In late 1993 registration for ISO 9001 will be submitted. A European site to manufacture prepreg will also be selected later this year. Early in 1994 the company will be moving to a larger facility in California.

Mr. Patz provided a brief history of the RS-3 resin since its initial development in 1988 for the Boeing SRAM II program (p. D-83).<sup>21</sup> The resin was licensed to Nippon Petrochemical<sup>22</sup> in 1989 and has since been qualified by Space Systems Loral, TRW, Composite Optics, and EDO. An F-15 radome flight article was built using an SF-5 Microply reinforcement in an RS-3 matrix by McDonnell Douglas.

Its chemical structure is shown on page D-84. The toughening agent is a submicron particulate that is dispersed in the RS-3. In response to a question from Mike Rigdon, Mr. Patz said the toughener is a co-polymer such as acrylonitrile or styrene. The chemistry for the cyanate materials is very simple: chemical reactions are the same at various cure temperatures—unlike the epoxy-based systems which undergo different chemical reactions at different cure temperatures. LtCol Obal asked if YLA was satisfied with design information that was available. Mr. Patz indicated that YLA works very closely with end users such as Composite Optics and TRW. In fact, Composite Optics is putting together "a wish list" for materials to replace the Amoco products. LtCol Obal then asked about the process control checks for batch material. Mr. Patz replied that if the raw materials are good quality and the process is in control there are no problems; if there is a problem it will typically be noticed long before the process is complete.

Mr. Patz presented some space-environment-related data on the RS-3-based materials. JPL exposed neat resin samples to electron radiation to determine changes in flexural modulus: there was essentially no change in modulus even after 10<sup>9</sup> rad (p. D-86).

<sup>21</sup> RS-3 is based on the Dow chemistry and was developed as a replacement for bismaleimide.

Nippon has generated a significant amount of data on RS-3. Mr. Patz held up a Nippon databook to show that it exists.

The toughness with the particulate is XX in.-lb/in.2, without the particulate, 0.35 in.-lb/in.2.

Outgassing data from Loral and Boeing show very low levels of TML and VCM (p. D-87). Moisture absorption properties are shown on pages D-88 – D-89.

A number of fibers have been utilized with the RS-3 to make prepreg for space applications (p. D-90): XN-nn-type fibers, high modulus pitch fibers ranging from P75 to P130, K139, the Japanese MnnJ-type fibers, UHM, and Kevlar. Unidirectional tapes of varying fiber areal weights have been manufactured: 27 gm/m<sup>2</sup>, 32 gm/m<sup>2</sup>, 70-80 gm/m<sup>2</sup>, and 150 gm/m<sup>2</sup>.<sup>24</sup> RS-3 seems to work quite well for the manufacture of thin prepreg plies although cost increases as ply thickness decreases: only 15 lbs/day can be made of the 32 gm/m<sup>2</sup> material.<sup>25</sup> XN-50- and Kevlar-reinforced fabrics of varying widths have also been made.

Resin development is a continuing activity. A replacement for Amoco 1962 is under development as well as 2 curing adhesive films and 2 improved RS-n matrix resins (p. D-91). Several of the commercially available cyanate esters were described (pp. D-92 – D-93): a replacement for the 1962 resin, REX 366 depends on the availability of the Dow material; Dk 2.75 and Dk 2.66 are Ciba Geigy's PC resins; the Allied Signal phenol triazine PC resin is believed to be a promising replacement for PMR-15;<sup>26</sup> the XU 1787 Dow materials are still experimental. YLA's matrix resins (p. D-95) include RS-1, -3, -5, -6, -7, -8, -9,<sup>27</sup>-11, -12,<sup>28</sup> and -14, all having somewhat different cure temperatures (200 °F to 350 °F) and varying service temperatures (up to 500 °F). YLA also offers a range of Microply syntactic films (p. D-96).

## E. MR. SCOTT UNGER, BRYTE TECHNOLOGIES

Bryte Technologies is a small manufacturer of high technology prepregs, adhesives, and resin systems. Market thrusts for its products include spacecraft, aircraft, electrically transparent structures, structural radar absorbers, and other specialty applications (p. D-98). In terms of cyanate esters Bryte believes it offers more prepreg, adhesive, and resin systems than any other company. Specialty product development and follow-on production of materials are another area of expertise. Manufacturing capabilities include hot melt prepreg production, hot melt cyanate ester adhesive production, long fiber

<sup>24 27</sup> gm/m<sup>2</sup> is equivalent to a thickness of 0.6 mils, 32 gm/m<sup>2</sup> to 1 mil, and 70-80 gm/m<sup>2</sup> to 2.5 mils.

<sup>25</sup> At 100 ft/lb this material has a fiber volume fraction of 0.62. Its cost is driven by the high fiber cost.

<sup>26</sup> Brian Gilmore remarked, however, that it is brittle and difficult to toughen.

<sup>27</sup> RS-9 is for high temperature use (500 °F).

<sup>28</sup> RS-12, a 250 °F curing polycyanate resin, is based on the Dow chemistry.

bulk molding compound production, and resin transfer molding and filament winding resin systems (p. D-99). Hot melt prepreg products include unidirectional tape up to 12 inches wide, <sup>29</sup> fabric prepreg up to 50 inches wide, non-woven prepreg up to 50 inches wide, and precision slit tow prepreg.

Bryte's premier cyanate ester material for space is EX-1515, a resin based on the Ciba Geigy monomer and toughened by an unidentified proprietary method (p. D-100). This resin cures at a relatively low temperature—225 to 250 °F—which reduces post-cure residual stresses. Jet Propulsion Laboratory (JPL) data from an unidentified test indicate that a P75/EX-1515 composite mirror structure has 10X greater dimensional stability than the 350 °F cure, Dow cyanate-based systems. Its excellent conversion level following cure assures microcrack, radiation, and solvent resistance. Moisture absorption and outgassing properties appear to be comparable to other cyanates. Relevant space applications include dimensionally stable space structures and optical benches, reflectors and solar arrays (using Kevlar woven fabrics), and other structures where outgassing, microcracking, and radiation are important concerns.

Bryte utilizes a number of fibers for both tape and fabric prepreg including the high modulus Mitsubishi, Nippon, and Amoco pitch carbon fibers and the Toray PAN fibers as well as Kevlar, fiberglass, quartz, and Spectra (pp. D-101 – D-102). A typical cure/post-cure cycle is illustrated on page D-103. Typical neat resin and laminate (XN-50A fiber) properties are shown on pages D-104 – D-106). Mitsubishi recently provided data comparing Bryte's EX-1515 with ICI's 954-3, both reinforced with K1352U pitch fiber (pp. D-107 – D-110). The EX-1515 matrix composites compared very favorably with the 954-3—and, in fact, the 0° tension, compression, and flexure strength data for the EX-1515 composites are measurably higher than those for 954-3. Microcracking data are listed on page D-111; relevant photomicrographs can be seen on pages D-112 – D-114.

Bryte Technologies also produces specialty adhesives—EX-1516 is the toughened cyanate ester-based adhesive (pp. D-115 – D-116). It meets the NASA outgassing specifications and exhibits superior electrical properties to the epoxy film adhesives. EX-1516 is available in unsupported or supported (with polyester, fiberglass, or quartz) films. EX-1502 is a toughened cyanate ester paste adhesive (p. D-117). It will meet

<sup>&</sup>lt;sup>29</sup> Cured ply thicknesses range from 1 to 1.5 mils.

<sup>30</sup> A free-standing post-cure treatment at 480 °F increases T<sub>g</sub> but apparently does not increase the residual stress levels.

NASA outgassing standards and has better mechanical and electrical properties than the corresponding epoxy adhesives. Both adhesives can be cured in the 250 to 350 °F temperature range.

Other cyanate ester technologies (pp. D-118 – D-119) include resin transfer molding and filament winding resins (e.g., EX-1510, EX-1530, and EX-1532); syntactic foam (EX-1541); other prepreg resins (e.g., BTCy series, EX-1505, and EX-1509); and other adhesives (e.g., BTCy-1B, EX-1537 series). Service temperatures for the RTM and filament winding resins range from 400 to 600 °F, for other prepreg resins, from 300 to 600 °F, and for other adhesives, 450 °F. A summary of all Bryte Technologies products can be found on pages D-120 – D-123.

Mr. Unger concluded his written comments by stating that Bryte offers a wide array of cyanate ester products to meet many needs. Since both resins and adhesives have been developed, they offer a complete/coordinated material system to designers. Bryte's EX-1515 resin, in particular, offers excellent properties with respect to dimensional stability and microcracking and radiation resistance. Bryte's lead times are relatively quick, on the order of 2 to 4 weeks, and Mr. Unger claims they are cost effective. He believes that Bryte Technologies is a reliable source of high technology materials, capable of meeting current, continually evolving programs.

## 4. COMPOSITE PARTS FABRICATOR PERSPECTIVES

Composite parts fabricators were asked to discuss their current experience with graphite-reinforced polycyanate composites, particularly material uniformity and processing-related issues. Results/data from any additional testing (including data as identified in Chapter 3) were also to be presented. In addition, they were asked to bring actual components/parts that have been fabricated using the graphite-reinforced polycyanate composites, especially those with which problems were encountered. Other discussion topics were to include suggested R&D directions and initiatives to fulfill their needs and requirements.

## A. MS. CHRISTINE BARKER, SPARTA

Sparta's efforts, supported by BMDO and directed by the Army, have been focused on polycyanate composites for interceptors. These resins are of interest for such structures due to their low moisture absorption, ease of processing, availability of high modulus fiber-reinforced prepreg, and higher operating temperatures following a post-cure cycle (p. E-2). Project objectives included evaluating the cyanate composites for the potentially low cost, matched metal net mold process and fabricating actual interceptor structures for full-scale tests.

YLA's RS-3 and ICI's 954 resin systems were utilized in this evaluation. Sparta validated YLA's moisture absorption data (p. E-3). As it turned out, Sparta was unable to use the YLA material: Nippon would not sell the fiber since an interceptor is considered to be a weapon system. Sparta found, in their experience making 6-inch long, FI700-reinforced<sup>1</sup> cylinders, that the consistency of the ICI 954-3 resin during cure was like water. Consequently, it ran out of the mold, resulting in resin-starved areas in the finished composites (p. E-4). Sparta now uses the 954-2A toughened resin, a controlled flow formulation of 954-3.

Sparta worked with one of the Ground-Based Interceptor prime contractors on the design of an exoatmospheric interceptor structure. On a tool trial, using a T300 fiber

Both Tonen's FT700 tape and fabric were used as reinforcement.

reinforcement, the first fabricated structure exhibited reasonably good resin quality: there seemed to be little fiber wash-out from the bulk. The tool trial GBI structure (with 60 percent T300 fiber) was passed around the audience for inspection. A GBI structure was fabricated for full-scale testing using an ultra-high modulus FT700 fiber. The structure was then fully loaded with dummy masses and tested under static and dynamic conditions as well as thruster loading conditions. It was able to meet the natural frequency structural load requirements. The baseline material for the GBI structure was titanium; a number of parts were needed to put together a complete structure. Ms. Barker mentioned that one of the goals of this program was to show that the cost of the composite structure was equivalent to that of the baseline structure; this was accomplished via parts reduction since the structure could be fabricated in essentially one piece. The optical bench for the interceptor is mounted as a separate structure; there is no housing for the optical components. She reiterated that the designs from which Sparta has fabricated structures are contractor-driven, with stiffness being the primary requirement.

Tool design and fabrication are important factors in keeping the cost of the matched metal mold process low. For only one or two parts, the net-shape molding process will not be cost effective; but for a hundred or more, it may be. LtCol Obal asked about tool deterioration over time. Ms. Barker replied that Sparta has fabricated over 1,000 parts for a shoulder launched missile application, so an experience base on tool wear does exist.<sup>2</sup>

Another BMDO-funded project is examining integral molding of a constrained layer visco-elastic material (VEM) with the structural composite for passive damping of an interceptor optical system (pp. E-5 – E-6). Sparta is using the matched metal mold process to fabricate this structure as well. The outer layer of the composite sandwich is slit to allow differential shear of the cylinder. The 3M AF-32 VEM has a cure cycle (350 °F) that matches that of the cyanate ester.<sup>3</sup> Some simple panels and cylinders were fabricated during the manufacturing development phase. Modal testing of the cylinders at 200 Hz showed 5 percent damping with the VEM, 3 percent without the VEM. The interceptor structure was being fabricated at the time of the cyanate workshop and was to undergo modal testing in July 1993. In response to a question from LtCol Obal regarding active piezoelectric sensors and actuators, Ms. Barker indicated that the cure temperature may have to be lowered to embed such devices into polycyanate composites.

Tool materials are selected and tool designs are tailored to meet a particular production requirement with minimal wear during that production.

At lower cure temperatures there would be a wider selection of VEMs from which to choose.

Ms. Barker concluded by saying that the cyanate resins appear to be attractive for these interceptor and missile applications (p. E-7). The material appears to process well and good quality, high modulus fiber-reinforced 954-2A prepregs and laminates have been obtained. Sparta has also been able to demonstrate that the VEM material can be molded in situ.

## **B. MR. JAMES KOURY, PHILLIPS LABORATORY**

Nir. Koury discussed fabrication activities at the Phillips Laboratory, which was responsible for providing the Clementine solar array panels. Phillips Laboratory uses an isogrid structural design, initially developed for fairings. The isogrid solar array panel design could be 15 to 30 percent lighter than a comparable honeycomb sandwich panel design, which is more typically used. He emphasized the low outgassing properties of the cyanate-based materials that makes them attractive for space applications (p. E-10). Mr. Koury then reviewed the available cyanate materials in terms of cure temperatures, glass transition temperatures, moisture absorption, applications, and lead times (p. E-11). In most cases the lead time was too long given the tight schedule for the delivery of the panels. The BTCy-1 resin was selected due to Bryte's rapid respond time. Properties of this resin can be found on page E-12. Mr. Koury indicated that cost was high: tow prepreg cost \$375/lb, unidirectional tape, \$250/lb.

Mr. Koury then described the manufacturing set-up. The isogrid mold consisted of a 3-ft by 5-ft isogrid rubber mold, 2 metal plates, a metal caul plate. The composite skins and ribs were laid up by hand, which was found to be faster than other approaches for making a few parts.<sup>5</sup> The composite skin and ribs were contained in a vacuum bag with the entire mold (p. E-13) and co-cured using the 350 °F cure cycle shown on page E-14.

The initial panel (IM7/BTCy-1) exhibited severe warping, 0.4 inches across the panel. This was apparently caused by thermal expansion differences among the rubber mold, Kapton,<sup>6</sup> and the composite;<sup>7</sup> in addition, the mold was unbalanced. Compression (p. E-15) and tension (p. E-16) properties were evaluated for this panel and were found to

<sup>&</sup>lt;sup>4</sup> McDonnell Douglas apparently fabricated 2-ft by 2-ft panels as a demonstration for the fairing application.

According to Mr. Koury the panels could also be filament wound.

The Kapton dielectric layer, 2 mils thick, is used to prevent the solar cells and their attaching wires from shorting out as a result of contact with the conductive graphite fibers.

The thermal expansion of the rubber is 140X greater than that of the composite.

be comparable to those measured by others. By using P75 fiber in the second panel, warping was reduced to 0.25 to 0.3 inches across the panel. In this case warping is believed to be due to rubber expansion. Ms. Burns asked if a lower cure temperature—250 °F—was considered, to which Mr. Koury replied that it was.

Mr. Koury concluded by saying that the panels were able to meet every requirement—frequency, mechanical, and physical properties—except flatness. Unfortunately this was a critical problem affecting attachment of the panels to the Clementine structure. With a metal barrier in the mold it was thought that 0.2 inches warping could be achieved. Mr. Koury also indicated that tow prepreg variability did not affect part quality. LtCol Obal noted that Phillips Laboratory had been asked to fabricate this structure on a short turnaround cycle and that a different structural design than is typically considered was selected. As a result of the warping problem, the solar array panels were fabricated by Composite Optics.

## C. DR. SURAJ RAWAL, MARTIN MARIETTA

Dr. Rawal began by stating, as other speakers had done, that the cyanates offer a number of advantages, a primary one being that they process like epoxies. The objective of Martin Marietta's efforts is to establish the required heritage/experience and user confidence so that the these materials can be inserted into real systems. Dr. Rawal believed that the Gr/PC composites offer increased system robustness and reliability.

Fabricated components and demonstration structures include flat panels, tubes, honeycomb sandwich panels, thermal management components, and the SAWAFE (Satellite Attack Warning and Assessment Flight Experiment) panels (p. E-21). Material procurement and process specifications are developed in-house. The procedures utilized by Martin Marietta for processing, NDE, machining, joining, and sandwich construction are outlined on page E-22. Either a vacuum bag/oven cure or autoclave cure cycle can be used to process the composite. The vacuum bag process is limited to 4-ft by 8-ft parts due to processing difficulties (porosity). Machining techniques include diamond band saws, laser, and waterjet cutting.

The SAWAFE panel, a flight experiment on the STEP-3 (Space Test Experiment Platform) mission, contains embedded radio frequency, laser, and X-ray detectors (p. E-23). The materials and construction of the panel are illustrated on page E-24. LtCol Obal noted the SAWAFE panel had requirements in addition to those for structural/load-bearing conditions: RF and charge build-up. Rich Lewis asked if the core-

filling foam was precured; Dr. Rawal replied that it was and that it was attached by secondary bonding. There were apparently some issues on coating adherence and bonding. Dr. Rawal also showed an example of a radiator (p. E-25).

Martin Marietta has generated material property data on selected composites using a range of high modulus fibers from Fiberite, YLA, Bryte Technologies, and Amoco (p. E-26). A few demonstration structures have also been fabricated and tested. During this experience some issues and concerns have arisen. Martin Marietta concluded that the vendor-recommended autoclave cure cycle yielded well-consolidated parts so there were no obvious processing problems. One of the concerns, however, was resin content inconsistency in some of the prepregs: across a one foot-wide sheet of prepreg resin content varied from 30 percent to 31 percent to 36 percent (p. E-27). LtCol Obal asked if this variation was normal or if the required parameters are tighter than what vendors typically provide. According to one of the material suppliers a typical range is ±3 percent, although ICI usually has a variation of ±2 percent. Edge-to-edge variations noted by Dr. Rawal, however, are considered abnormal. Dave Powell indicated that the resin content test is not very sensitive. Dr. Rawal noted that prepreg tack was sometimes very low but could not quantify it. He indicated in response to a question that Martin Marietta spoke with the vendor, ICI, regarding this problem. Dave Powell stated that Martin Marietta had been informed ahead of time that there was a problem with the prepreg. While ICI has an extensive quality control system, active/intelligent control is not used: the spacecraft user market does not call for it. It may be a material maturity problem as is often observed for advanced materials that are not available in large, commercial quantities. Nonuniformities in polycyanate film adhesives were also observed. Prepreg material supply/availability is a continuing problem (p. E-28). The recent scare with the Dow resin represents a major issue for those contractors who have evaluated and qualified that material. There is interest in knowing the resin supplier perspective of the situation. As is the case for other advanced materials, the ever-changing resin formulations represent a problem to contractors in terms of data collection and material qualification. It greatly influences designer confidence and use of the materials.

According to Dr. Rawal, perceived or real toxicity concerns during cyanate matrix and prepreg processing need to be evaluated: we do not want these composites "to become the beryllium of thermoset composites." According to Dave Powell, the dangerous part of the process is associated with cyanation and is over when the resins are received. Pam Burns inquired about the safety of the cyanates with respect to the epoxies.

Mr. Powell indicated that there had been no incidence of dermatitis to his knowledge but could not unequivocally conclude that the cyanates were safer than the epoxies. Safety issues are a concern for the Lonza facility, where the cyanation step is carried out. The gases produced there are the same as the Bhopal gas: it is bad stuff which they have been able to handle so far. Mike Rigdon asked if Lonza would run into environmental problems in the future such that they might be required to shut down the facility. At this point in time, no one knows. Another attendee inquired about waste disposal. The response was that it was not any more of a concern than that for epoxy prepregs. Scrap material could be disposed of in the cured state or left uncured. In the latter case it may have to be treated as hazardous material.

Dr. Rawal concluded his presentation by describing topics for further research and development (p. E-29). The four general categories are structure-property relationships, design databases, processing, and joining and assembly. Investigations of additives for toughening and matrix studies of microcracking resistance are desired in the area of structure-property relationships. Of particular interest in the area of design data are fatigue life, fracture toughness, and damage tolerance. Knowing the additive-rheology-tack-processing window would be an advantage for determining optimum processing conditions. Low cost, intelligent processing schemes are also needed. Adherence of environmental coatings seems to be an issue for joining and assembly operations.

#### D. MR. GARY KRUMWEIDE, COMPOSITE OPTICS

Composite Optics, Inc., has extensive experience with a variety of polycyanate materials systems. Reinforcements include P75, K149, XN50, XN70, and T300; matrix materials include 954-3, ERL 1999, EX-1515, and RS-3. (pp. E-31 – E-32). Though five material systems have been evaluated, only three were discussed. Fabricated hardware includes 4 telescopes, 3 bezels, 11 solar array panels for Clementine, 5 reflector parts, 3 mirrors, 2 phased arrays, 10 feed horns/waveguides, and 10 MUX cavities. Lots of coupon tests have been performed. B-allowables were established from that coupon data. Data on other less typical properties such as EMI and ESD are just now in the process of being evaluated. Thin-ply specimens were shocked at liquid nitrogen temperatures and no microcracking was observed. Joints have been bonded and thermally cycled using no fasteners. Mike Dean commented about the difference in evolved contamination products between epoxy and cyanate systems. In response to an inquiry from Mike Rigdon, Mr. Krumweide indicated that all the listed materials had been used to build components/

structures. For the telescope application the choice of the 954-3 resin was dictated. For the Clementine structure the prepreg material was furnished. Some of the honeycomb sandwich panels were co-cured; no problems were noted, although the speaker indicated there was a trick to it.

Mr. Krumweide discussed general traits of the cyanate prepregs in terms of processing (p. E-33) and mechanical (p. E-34) and physical (p. E-35) properties. General processing traits of the cyanates are similar to the epoxies. Lay-up characteristics are as good or better than those for epoxies. Flow during cure is higher and requires special attention to bagging. Differences in tack are slight.

Mechanical and physical properties are generally quite similar (pp. E-36 – E-38). Quasi-isotropic laminate properties such as tension modulus and strength and compression strength and micro-yield properties are essentially equivalent to those of the epoxy composites. The cyanate systems have a high resistance to microcracking<sup>8</sup> (p. E-39), and compression and shear modulus properties appear to be unaffected by radiation levels up to 200 million rads. Interlaminar properties are slightly lower than those of modified epoxies such as ERL 1962. Mr. Krumweide emphasized the importance of defining a test plan in an intelligent manner. He also stated that resin-dominated properties may be lower than those for the same epoxy laminates.

The glass transition temperature (Tg) is determined by the post-cure temperature; according to the speaker, more work is needed in this area. Thermal expansion values for isotropic cyanate laminates are slightly more negative than for the same epoxy laminate, although the CTE is linear over a greater temperature range (p. E-40).<sup>9</sup> In response to a question from Pam Burns, Mr. Krumweide stated that many samples taken from locations all over a composite panel were used to obtain CTE numbers. The amount of absorbed moisture, affected by Tg, is about 1/3 that of the same epoxy laminates, and changes as a function of temperature (p. E-41) and humidity (p. E-42).<sup>10</sup> It is, however, absorbed (and desorbed) at a rate 5 to 10X faster than that of the same epoxy laminates. CME values are generally the same as for epoxy composites but the associated strain is about 1/3 that

The amount of microcracking has not been quantified. Specimens are sectioned and examined for cracking via optical microscopy. Below some level of microcracking no changes can be observed in the material.

This means there is no heating/cooling hysteresis. Thermal expansion can be adjusted via the addition of resin. The data on page E-40 is from coupons from 3 locations on a 1.11-inch diameter tube.

Moisture absorption is measured on saturated laminates.

measured in the epoxy composites due to lower overall moisture absorption (see p. E-43 for data on a prototype mirror structure).

Composite Optics, Inc., has evaluated joint properties: butt tension, butt shear, and lap shear (p. E-44).<sup>11</sup> Bondline allowables for the cyanate composites are lower than those for a similar epoxy composite. Moisture content can influence bond strength. When COI is co-curing Kapton and cyanate composites, the structure is held overnight under vacuum to reduce the moisture content. Dave Powell asked if this moisture/bond strength relationship was observed with Kevlar cores. Mr. Krumweide replied that it was not because the cores are dried first. If not, carbonates form and a molecular bond will not occur. Dave Powell mentioned that these carbonates can decompose to form pores at 400-450 °F.

Mr. Krumweide mentioned several other characteristics of the cyanate composites (p. E-45). He indicated that the cured cyanate composites offer good solvent resistance. Though the resin modulus is lower than that of an epoxy its strain to failure is higher. The cyanates are more resistant to ultraviolet radiation and exhibit better long-term thermal stability. These resins cost more and are in limited use at the present time. Concerns (p. E-46) include a post-curing effect which needs to be verified and inconsistent moisture data.

He concluded his presentation by describing the fabrication of the Clementine solar array panels. Materials and parts utilized in panel construction include skins and doublers, honeycomb core, Kapton film and tape, adhesives, inserts, and potting compound (p. E-47). The Kapton film was co-cured with the cyanate composite skins, Core filling was completed after secondary bonding of the skin to the core; panel edges were closed with Kapton tape. Inserts were bonded after the rest of the panel was completed and doublers were secondary-bonded to the outer surface. Actual measurements of flatness, parallelism, weight, and insert pull-out strength were well within the required values (p. E-49).

#### E. MR. RICH LEWIS, TRW

TRW's initial interest in polycyanates was for reasons of low moisture expansion. TRW has been investigating these materials since 1988. A wide variety of reinforcements have been used including the Mitsubishi K-series, Dupont E-series, Amoco P-series, and

<sup>11</sup> The joints were bonded using a Hysol 9394 adhesive.

Nippon XN-series fibers, and YLA microply syntactic foam (p. E-50). Outgassing and mechanical properties have been characterized. Outgassing performance was measured in terms of TML and VCM for a number of materials such as RS-3 and 9054-3 neat resins and RS-3 with various fibers (p. E-52); measured data are well below the acceptance values. Mechanical properties of the Gr/PC composites are, in most cases, equivalent to those of the Gr/Ep composites. Acceptable bond strengths have been measured using film or paste epoxy adhesives.

Processing-related characteristics were also described (p. E-54). The low viscosity of the cyanate resin makes it attractive for debulking and wetting out fiber bundles; it also reduces fiber bridging. Mr. Lewis commented that tack is a function of the temperature: there is apparently a temperature window in which the tack is the right level for lay-up. It was suggested that ice crystals should be kept off material stored in a freezer. When possible the material should be kept in a vacuum-sealed, moisture-resistant film bag to avoid moisture absorption. The characteristics of the cyanate resins also make them somewhat more amenable to production of thin tapes, ~0.5 mils.

TRW's initial fabrication experience was on the unclassified HARD (High Accuracy Reflector Development) program (p. E-55). Ultra-thin, 130 Msi modulus prepreg (<1 mil/ply) was used to make a thin sandwich membrane (<15 mils total thickness). The reflector, illustrated on pages E-56 – E-57, was 21 feet in diameter. All of its panels were curved and the center one was coated with aluminum.<sup>12</sup> The backup structure for each panel was an isogrid (p. E-58). In reference to Jim Koury's presentation, Mr. Lewis remarked that TRW used hard tooling rather than trapped elastomeric tooling. He commented as an aside that tooling was key to successful fabrication. And this was the part of a program on which program managers were most likely to skimp.

TRW has fabricated structures for ground and flight experiments. The Smart Strut was an early effort on the ACESA (Advanced Composites with Embedded Sensors and Actuators) program (p. E-60). The AMASS (Advanced Materials Applications for Space Structures) solar array structure was built for a ground experiment (pp. E-61). ACTEX II (Advanced Control Technology Experiment) is scheduled for the STEP-3 flight in July 1994. It is fabricated in an autoclave from T300/RS-3 tape laid up by hand (p. E-62). Overviews of the passive damping/active control experiment and the final solar array yoke

 $<sup>^{12}</sup>$  The dots on the dark panels were used for measurement purposes.

design are shown on pages E-63 – E-64. LtCol Obal commented on the idea of building electronics into the structure. Efforts to miniaturize sensors, actuators, and control and processing electronics enable fabrication of such integrated structures. TRW's efforts have been addressing some of the issues in this and other programs. LtCol Obal also recognized the management of high voltage in conducting materials as an important contribution.

TRW has also fabricated integrally hinged, segmented boom structures (p. E-65). The sections are 7 to 8 inches in diameter and are transitions from round to square cross-sections (p. E-66). Point design tests have been performed. A wide variety of tool materials and mold releases have been used: so far no difficulties have been noted.

Data on space environmental effects are being obtained through a variety of space tests (p. E-68): EOIM-3 (Evaluation of Oxygen Interactions with Materials); LDCE-4, 5 (Limited Duration Candidate Materials Exposure); and Matlab 1. These short-term, recoverable experiments are mainly concerned with atomic oxygen effects.

Mr. Lewis concluded his presentation with some general comments and issues. TRW is interested in having several suppliers rather than a single source for a particular material. In addition, TRW would like to see lower resin contents in ultra-thin tapes in order to reduce the amount of debulking that must be done. Better equipment for prepreg manufacturers to make thin ply prepreg is desired. Brian Gilmore stated that the volume of business for 1-mil or 0.5-mil thick tapes/prepregs is rather small for larger structures even though it may be enabling for more esoteric applications. LtCol Obal asked if such prepreg machines existed. The response was that the processing people know what to do to make thin plies. However, as an example, the backing paper is stronger than the ply and is a limiting factor in the manufacture of thin plies: a low CTE paper would, therefore, be desirable. YLA can make thin tape in small production runs and has experienced no physics or engineering difficulties. Gary Patz did state that the rheological properties of the resin were important—the proper formulation is necessary. Another attendee remarked that since the markets for thin plies are essentially small, niche markets it makes more sense for a small company to invest in them than a large company. Pam Burns commented that the cost of the thin-ply prepreg materials is higher but Mike Dean observed that such materials were enabling for some applications.

#### F. MR. HARRY DURSCH, BOEING

Mr. Dursch described several Boeing programs, including the ACESS (All Composite Experimental Spacecraft Structure) program, that are considering Gr/PC

composites. Flat laminates and other shapes have been fabricated from cyanate ester resins (YLA's RS-3 and ICI's 954-1, -2A, and -3) typically reinforced with quartz or fiberglass (p. E-71).<sup>13</sup>

In the ACESS program (p. E-72), Boeing and subcontractor Ball Aerospace are to design and build an all-composite bus structure for STRV-2 (Space Test Research Vehicle), an international space flight experiment to be flown in 1995 (p. E-73). System level design input is being provided by the Naval Research Laboratory who will also perform the qualification testing for the completed structure. The schedule (p. E-74) is an aggressive one with delivery of the structure 14 months after program initiation. Boeing is allowing 6 months for screening candidate materials, developing process and material specifications, and developing the necessary lamina, sandwich, and insert design data.

Technical objectives are to demonstrate lower weight and cost and greater dimensional stability than an aluminum structure. The baseline design (pp. E-75 – E-77) is a hexagonal box with removable side panels. Since reduced part count reduces cost, Boeing is using a co-curing process to fabricate and assemble the structure (p. E-76). Part count for the envisioned structure will be reduced by 50 percent and complex assembly tools will be eliminated. With 15 percent contingency, the composite structure is expected to weigh 25 lb, 10 lb less than the aluminum one. Such a fabrication/assembly approach may allow for increased design flexibility, too. According to Harry Dursch, fabrication and assembly of the structure using the co-curing process will require 1,000 hr; a more traditional approach involving secondary bonding would require 2,150 hr. The speaker showed a co-cured hexagonal panel (fabricated from a cyanate ester composite under an IRAD project) similar to a deck structure mounted inside the ACESS bus walls.

Important material parameters for this structure include dimensional stability, outgassing, CTE, processability, thermal and electrical conductivity, strength and stiffness, and space environment survivability (p. E-79). The cyanate ester appears to meet the requirements although its flexural modulus is lower than either the epoxy or the toughened epoxy (p. E-80). Resin viscosity is an important factor in making a good quality, co-cured part. Four cyanate resins having a wide range of viscosities are being investigated: RS-3,

Over 3,000 lb of cyanate ester prepregs and adhesives have been utilized. Boeing developed an extensive database on the quartz/954-2A material for use on large diameter radomes. However, due to environmental restrictions on the use of methylene chloride, 954-1 will not be available on a long-term basis. Boeing had planned to use quartz/954-2A for the 26-ft diameter MILSTAR radomes but was redirected to use Gr/Ep due, in part, to the Dow resin supply problem (which has since been resolved).

<sup>14</sup> Those times do not include tooling, manufacturing, or quality assurance.

RS-3M, 954-2A, and EX-1515.<sup>15</sup> Typical cure cycles for 2 resins are shown on pages E-82 – E-83: temperatures vary as a function of position, i.e., tool side or bag side, and time. Due to the unique combination of properties found in the cyanate composites they have been selected as the baseline material (p. E-85). Co-curing the cyanate composite and the aluminum core is believed to be a moderate risk. Based on an earlier NASA program, Boeing has in-house capabilities to apply aluminum foil to composite structures for protection in the space environment should the need arise.<sup>16</sup> One of the attendees asked if aluminum foil was necessary for grounding. Someone replied that such a requirement may no longer exist since apparently sufficient electrical conductivity is available for ESD and grounding.

Mr. Dursch identified several other programs in which Boeing is examining cyanate composites (p. E-86): Pluto Fast Flyby (JPL), Integrated Power Panels (JPL), Composites Structures Assembly (Classified), Low Temperature Structural Carbon Fiber Prepreg (Boeing Commercial), and Composite Joining Technology Development (PL, p. E-87). Ms. Burns inquired about the number and type of joint tests performed. Mr. Dursch indicated that Boeing is testing a variety of polymer composites and various adhesives. Lap shear tests and surface preparation techniques are being evaluated.

Harry Dursch concluded by reiterating Boeing's experience with the design, fabrication, and testing of the cyanate composites. The epoxy-like processing behavior of the cyanates make them attractive for the described applications. The availability of the Dow-based cyanate resins was identified as a particular concern.

## G. DR. JOHN TRACY, MCDONNELL DOUGLAS

Dr. Tracy began by discussing the properties of fibers that have been investigated by McDonnell Douglas for space applications, in particular, strength vs. modulus (p. E-90) and elongation vs. modulus (p. E-91). The ultra-high modulus fibers, needed to make stiff, lightweight space structures, typically have low elongations which result in manufacturing difficulties. He then identified four applications in which these fibers have been considered (p. E-92): Single Stage-to-Orbit (SSTO) Rocket Technology, 17 Neutral

Other resins under consideration include 977 and 934 epoxies (p. E-84). Reinforcements of interest include P100 and K1100 fibers and T650 and IM7 fabrics. Several different aluminum honeycomb cores are being examined as well.

<sup>16</sup> These 2-mil thick foils are applied for atomic oxygen protection and for thermal control purposes.

<sup>17</sup> For both this program and the NPBSE, prepreg availability was a concern.

Particle Beam Space Experiment (NPBSE), Mobile Remote Manipulator Development Facility, <sup>18</sup> and the Ground-Based Surveillance and Tracking System (GSTS). <sup>19</sup> Note that some of these structures are quite large.

The fuselage space truss for the DELTA Clipper (single stage) structure is shown on page E-93. McDonnell Douglas fabricated a subscale square truss joint representative of those in the SSTO truss in an IRAD effort (p. E-94). The tubes were 9-inch squares; McDonnell Douglas experienced some difficulties in placing fibers around corners of the tube. An attendee asked if the tubes were required to be square. Dr. Tracy replied that it was not a requirement but it was usually done for ease of attachment. Both the truss member and a common truss joint were to be tested; a local buckling failure mode was predicted for the truss joint though the structure has not been tested yet.

GSTS telescope components were briefly described (p. E-95). The gimbal is the heaviest component in the sensor system that currently has a very narrow mass margin (p. E-96). An advanced composite gimbal is projected to weigh 30 to 40 percent less. On this program McDonnell Douglas planned to design, fabricate, and test a high modulus fiber-reinforced composite ring, trunnions, base plate, and support struts. Weight reductions for these parts (p. E-97) were projected to be 37 percent (30 g re-entry load) or 45 percent (10 g launch load, no re-entry). McDonnell Douglas was also involved in a now-cancelled program to build the GSTS thermal isolator. John Tracy indicated that his program management is sold on the cyanate composite materials. Pam Burns asked if McDonnell Douglas had any data at cryogenic temperatures. Dr. Tracy replied there was some data but did not provide specifics. He closed this part of the presentation with a chart showing the advantages of these materials for the McDonnell Douglas applications (p. E-98).

General advantages of the cyanate esters were described (p. E-99): low moisture uptake, low outgassing, excellent dimensional stability, high toughness, high T<sub>g</sub>, and low dielectric loss tangent. McDonnell Douglas has evaluated vendor data on several neat cyanate resins: 954-2A and -3 and RS-3 (934 epoxy for comparison). Evaluated properties include tensile strength, modulus, and strain, moisture uptake, T<sub>g</sub>, density, and outgassing and are shown on page E-100.

The 55-ft arm, Option B for the Space Station, was to be constructed from 14-in diameter tubes with 1-inch thick walls. John Tracy's group was told to use P120 fiber for reinforcement.

In the joint GSTS program with Hughes and Composite Optics, still existing at the time of the workshop, outgassing and dimensional stability were two major concerns.

Hybrid<sup>20</sup> and pseudo-isotropic<sup>21</sup> laminates have been tested as well (p. E-101). Specific coupon tests include 0° tension and compression, in-plane shear, short-beam shear, CTE, bolt bearing, and limited physical properties (p. E-102). John Tracy presented a significant amount of comparative data. A list follows: moisture uptake for hybrid laminates (pp. E-103 – E-104; compression strength (p. E-105) and modulus (p. E-106) of hybrid laminates; in-plane shear strength (p. E-107) and modulus (p. E-108) of hybrid laminates; short beam shear strength (p. E-109) of hybrid laminates; and tension vs. compression modulus (p. E-110) and strength (p. E-111) for hybrid laminates. Data on pseudo-isotropic laminates was summarized in a table (p. E-112). Pseudo-isotropic laminate tension strength (p. E-113) and modulus (p. E-114) data were shown as a function of the corresponding compression data. A question was raised about the suitability of the compression modulus test. Dr. Tracy replied that the trends were repeated using the same matrix with a different fiber: the modulus value was always higher using ASTM D695 than that using ASTM D3410A. Though tensile strengths are higher, tension and compression moduli and compression strength of the cyanate composites are typically lower than the comparable epoxy composites. Jim Koury commented that PL was unable to get good translation of properties. He believed it was due to material damage occurring in prepreg manufacturing. Dr. Tracy noted that the P120/RS-3 and P120/954-2A composites are currently undergoing evaluation on the Mobile Remote Manipulator program.

The truss member test and fabrication study was described in more detail (p. E-115). Both the hybrid composites and pseudo-isotropic laminates are being evaluated. Fabrication processes of interest are filament winding and the conventional hand lay-up method. The fabricated truss members are then tested in compression. Materials include P120/1939-3 with IM7/8551-7A, K139/977-2 with AS4/3501, FT700/PRS, and FT700/934 (p. E-116). The low viscosity of the 954-3 resin resulted in a poor quality part: wrinkles caused by about 10 percent loss of resin were evident in the cured part (p. E-117). Using a resin with additional thickening agents or using tooling with integral resin flow dams may solve the problem. ICI's 954-2A, a possible solution to the resin flow problem, has a higher viscosity and is more difficult to prepreg (p. E-119). It also has a slightly

Fiber/resin combinations include FT700-T800/934 or 954-3, K130-T800/934 or 9954-3, and P100-T650/ERL 1962 (0° fiber - off-axis fiber/resin).

<sup>&</sup>lt;sup>21</sup> Fiber/resin combinations include FT700/934 or 954-3, K139/934 or 954-3, K13B/977-2, and P120/ERL 1939-3.

higher moisture pick-up though it is still 3X better than that of epoxy. Dr. Tracy showed the recommended RS-3 autoclave cure cycle (p. E-120). McDonnell Douglas is interested in nonautoclave curing and investigated some unidentified processes for epoxy composites on a cryogenic tank program.

Dr. Tracy concluded by summarizing the advantages and disadvantages of the cyanates (p. E-121). The advantages had been identified by a number of other speakers. Disadvantages were thought to be lower compression strength and modulus as well as viscosity. When asked if he thought the differences between the epoxy and cyanate compression properties were statistically significant, Dr. Tracy replied that he did not know.

#### 5. DISCUSSION AND CONCLUSIONS

#### A. DISCUSSION

Closing comments were brief due to the late hour. The Dow resin availability problem was identified as a major concern. LtCol Obal closed by asking what should be done with the limited, available resources. A few comments were made regarding the database issue. Past database products have included a thin set of typical properties for a number of materials that were thought to be interesting. It seems clear, at least to the designers, that some sort of data book/document is needed as a rallying point for getting advanced materials into real systems. The Ketema-developed "Space Materials Selection Guide," funded by the M&S program, was cited as one of the few data books containing information on current advanced materials. It has been widely used by designers since little data are available elsewhere. Pam Burns indicated that the Ketema guide should be updated. Mike Dean suggested that, if the book were updated, the processing section should be reduced. Another attendee suggested that more credibility should be given to material supplier- or vendor-generated data. There is general agreement that documentation of data relevant to spacecraft applications is something the M&S Program could promote.

#### **B. SUMMARY**

All the participating groups were asked to identify information required on other groups to ensure utilization of advanced materials in real systems. That information is contained in this section. A number of other important points and issues identified by the participants throughout the workshop are summarized here as well.

## 1. Designers

Material selection for a particular design begins with a downward flow of design requirements. A lengthy list of design requirements ranging from overall system features to specific manufacturing concerns was provided by the speakers. The most important considerations were identified by a number of designers as program cost and schedule. Other important system goals may include, among others, spacecraft mission and lifetime; sensor instrument and/or payload performance; weight, particularly with

respect to launch vehicle step function costs; dimensional stability and rapid pointing and tracking capabilities; deployability; survivability; structural loads; radiation shielding; and thermal management. Manufacturing-related requirements important in the selection of materials include system reliability; availability and manufacturability/producibility of the materials, components, etc.; fabrication, assembly, and inspection costs; ease of assembly and repairability; worker safety/health concerns; and material handling. Processability, flight heritage, and previous, relevant experience with similar materials and applications are also considered to be among the most important factors affecting use of advanced materials in a real system. According to Mr. Cooney, flight heritage is a "religion" for designers.<sup>1</sup>

Once a balanced allocation of requirements has been determined, various materials can be investigated for potential use via trade studies. These trade studies should be as realistic as possible using acceptable design and analysis procedures. A system engineering approach is preferred for evaluating various materials for an application. System impacts and subsystem interactions need to be carefully analyzed. Results from a well-planned test program should be correlated with results from the analytical studies; such testing may include coupon tests to generate basic material property data and structural component and joint tests to demonstrate system performance. According to Dr. Dragone, small companies rely more on test than analyses. Proven process specifications and demonstrated producibility make the successful application of an advanced material more likley. It is also more likely that an adequate material property database will exist for such a material. Based on comments made by several of the designers, it appears that the perceived risk of using a material is less if there is a large database, such as for aluminum. Such a database includes information on fabrication and assembly experience (processes and cost), as well as material property data. In small companies, off-the-shelf materials are more likely to be used since the material property and manufacturing database already exists.

General material requirements to meet system requirements include high specific strength and stiffness, good compression modulus, adequate low cycle/high amplitude fatigue life, low molecular/moisture outgassing, low matrix microcracking, low thermal expansion, high thermal conductivity, and toughness for handleability during fabrication

It is not clear why contractors place so much emphasis on flight experience. Unless the structure is instrumented there is no good comparison of actual structural performance to design prediction: all that is known is that the "gee whiz" electronics and/or sensor systems work. The structure may be overdesigned by a significant margin that cannot be determined by an "it works" or "it doesn't work" approach to design.

and assembly. Other requirements that were mentioned include good electrical conductivity, good vibrational damping, low moisture strain, and creep resistance.

Material qualification tests vary from company to company. Space Systems/Loral evaluates composites via tests of mechanical and physical properties as follows: tensile strength, modulus, and Poisson's ratio; compression strength and modulus; thermal cycling (-180 to +135 °C, 10X, 1 lot minimum);<sup>2</sup> environmental tests from -100 to +100 °C; radiation; outgassing; CTE and CME; void content; and laminate density. Resinrelated processing features are also evaluated. Hughes qualifies materials in three different stages. The first stage is coupon testing via ASTM procedures to develop the design database: two different prepreg lots, 16 coupons for B-allowables.<sup>3</sup> Mechanical property tests include tensile strength, modulus, and Poisson's ratio; compression strength and modulus; in-plane shear strength and modulus; interlaminar shear; and flexural strength and modulus. Adhesion is evluated by lap shear and flatwise tension tests. Physical properties of interest are outgassing and volatile content, moisture absorption and microstrain as a function of humidity, CTE, electrical and thermal conductivity, density, fiber volume fraction, resin content, void content, tack, shelf life, Tg, resin flow, and gel time. System level tests, the second stage of qualification, are specific to the application and may involve fabrication and test of sub-elements or full-scale prototypes. These tests include bearing tests, flexural strength and modulus for honeycomb panels, thermal cycling (typically -40 to +80 °C, 24X for internal space structures), and dynamic loads. If the intended use is a critical application, a full-scale assembled part is tested to failure. Component/ subassembly functional tests and system level vibration and thermal/vacuum tests appear to be common space qualification tests for most companies.

Several important issues were raised relative to cost and manufacturing. A multi-build assumption is sometimes used to show that the cost of a composite structure can be lower than that of a metal one: composite structures typically only become cost effective beyond three to five units. It remains unclear that such an assumption is realistic given the current DoD and NASA budget environments. However, as several speakers indicated, part count reduction can contribute to reduced composite structure cost. The ability of a structure to perform more than one function—e.g., thermal control, EMI shielding, and

The 0° compression strength and modulus of thermally cycled specimens is measured at room temperature.

TRW tests 29 coupons from three prepreg lots. If only 16 coupons are evaluated, an additional knock-down factor must be applied.

support of mechanical loads—may offer cost advantages and should result in reduced system weight as well. The amount a program manager is willing to pay for reduced weight, though, depends somewhat on the launch vehicle cost increment.

The cost of tooling to make composite structures was mentioned since it is not often included in cost analyses. Data presented by Mike Dean on fixed wing aircraft showed that it represents about 7 percent of the total composite structure cost: fabrication, assembly, and inspection remain the high cost manufacturing items. One attendee commented, however; that material selection and design drive those high-cost items. For spacecraft, tooling costs may be higher if only one or two structures are built and since spacecraft designs and components may differ substantially from one program to the next.

The materials themselves—resins, fibers, and prepregs—were discussed at some length in terms of availability/maturity and quality, two issues facing all advanced materials. High performance materials are usually expensive because the market is small and, therefore, fabricated quantities are small and vice versa. Inconsistent quality of advanced polymer composites results from continual tweaking of matrix resin formulations from batch to batch. This, combined with the continual movement of suppliers and materials (resins and fibers) in and out of the market make it difficult for a user to fully evaluate and qualify a material for application in a real system. The supply of high-quality, high-modulus, domestic pitch fiber appears to be an issue. A recent public law requires that a minimum of 75 percent of pitch carbon fiber must be procured by DoD from domestic sources. A similar law applies to the PAN carbon fibers (50 percent). Though these requirements can apparently be waived, there seems to be limited experience in obtaining a waiver. Several suppliers and fabricators mentioned the good handling characteristics of the foreign fiber during manufacturing. Thin ply prepregs with high modulus graphite fibers, of particular interest to the spacecraft community, are not available. The missile community is interested in high modulus fibers, too, but would like higher strength. The PC composites are not deemed enabling although the fact that polycyanates can be treated like epoxies is a processing advantage. The potential for increased robustness and reliability may make them somewhat more attractive to program managers, however.

## 2. Material Suppliers

Material suppliers provided information on what they needed to know from both designers and fabricators to ensure adequate material development. Information required

from designers includes mechanical property goals; dimensional stability limits; expected service temperature; environmental severity of thermal cycles, radiation dosage, atomic oxygen exposure; and minimum allowable outgassing. The particular design approach—laminate or sandwich—and preferred joining techniques—co-curing, adhesive bonding, or fasteners—need to be defined for the supplier as well. Specific material features to be determined by the designer include selections of pitch or PAN fiber (for stiffness and thermal management) and tape- or fabric-based prepregs, fiber volume fraction (or fiber areal weight) and resin content, and cured ply thickness.

According to Dave Powell, material suppliers need processing-related information from prepreg manufacturers and part fabricators. From prepreg manufacturers such knowledge includes raw material reactivity, supply stability, mixed resin viscosity and reactivity, pot life/shelf life, fiber wet out, sizing compatibility, and environmental issues. From fabricators it includes acceptable cure cycles with respect to temperature and pressure limitations of user facilities; flow control and bleed schemes; prepreg out-time; tack and tack life; prepreg drape and radius conformance; and tool release. Effects of post-cure residual stresses must be evaluated and, if necessary, resolved from both a design and fabrication/assembly perspective. Repair issues must also be addressed. Cost issues remain an overriding concern for the material suppliers and their customers.

All the material suppliers provided a significant amount of data that will not be discussed here. Dave Powell described Imperial Chemical Industries' characterization program. Tension, compression, shear, and flexure properties, fracture toughness, edge delamination, thermal cycling and microcracking, and solvent resistance are usually evaluated. Additional tests could include moisture absorption, thermal expansion, density, and other physical properties. These tests appear to be typical of those performed by material suppliers. Just as an example, tensile strength, modulus, and strain are measured at room temperature for 0° and 90° fiber orientations; open hole tensile specimens with 25°/50°/25° lay-ups are tested at room temperature with strength and modulus being evaluated under 180–300 °F wet conditions.

Costs of the PC prepregs are not greatly different from those of the epoxy prepregs. Resin costs appear to be about the same. In the advanced composites, prepreg costs are driven by the costs of the high performance fiber. Pam Burns indicated that, although composite material (and component) costs can be high, overall systems costs can be lower if the advantages and flexibility of composites are utilized in the design, i.e., via parts reduction. John Tracy commented that McDonnell Douglas was able to show composite

part cost can be lower. Sparta was able to show that the cost of a composite GBI structure fabricated by their net shape molding process was equivalent to that of the baseline titanium structure via parts reduction. Boeing has predicted substantially reduced weight and fabrication and assembly time due to a 50 percent reduction count in part for ACESS.

### 3. Fabricators

Component fabricators are naturally interested in processing-related features of the composites in terms of resin characteristics (e.g., chemistry, viscosity as a function of temperature, T<sub>g</sub>, resin content); fiber/prepreg characteristics (e.g., limitations on drape and radius conformance, tack and tack life, out-time); cure cycles; and post-cure residual stresses. Rich Lewis noted that prepreg tack is a function of temperature and there is a temperature window in which tack levels are at the right level for lay-up.

Fabricators also described their experience working with PC composites. The typically low viscosities of some of the PC resins during cure were mentioned by a number of speakers.<sup>4</sup> For fabricators this means that special attention must be paid to bagging and bleed schemes as well as tools.<sup>5</sup>

Tool design and fabrication are particularly important for the near net shape molding process described by Christine Barker. Tools are designed and fabricated to provide minimal deterioration over the intended production run. Tool/mold design was a critical factor in the Phillips Lab Clementine program: the isogrid solar array panels were not able to meet the flatness requirements as a result of thermal expansion differences between the rubber isogrid mold, the Kapton, and the composite. The panels were successfully fabricated by Composite Optics using a different structural design. Rich Lewis commented that tooling was key to successful fabrication but was the part of a project on which program managers were most likely to scrimp.

Martin Marietta noted some inconsistencies in prepreg resin content ranging from 30 percent to 31 percent to 36 percent across a 1-ft width. This is considered abnormal and may be a material maturity problem. Dave Powell stated, however, that the resin content test method is not very sensitive. Inconsistencies in prepreg tack (apparently one lot only)

Controlled flow resins are available a la Imperial Chemical Industries' 954-2A. Rich Lewis noted, however, that low viscosity is good for debulking and wetting out fiber bundles and reduces fiber bridging.

One speaker noted that temperature in the curing composite varies as a function of position—tool side vs. bag side—in an autoclave. Since viscosity varies as a function of temperature, process conditions will have to be carefully controlled to minimize through-thickness variations in the cured part.

and polycyanate film adhesives were also noted. Apparent coating adherence and bonding issues were raised by several speakers as well. Difficulties in going around corners and low compression strength and modulus properties can probably be attributed to the fiber.

As for designers, changing resin formulation represents a problem. In addition to the difficulty of collecting data and qualifying material, slight changes in resin formulation may significantly affect processing conditions. Availability of thin ply prepregs containing high modulus fibers was also identified by the fabricators as an issue. TRW is interested in ultra-thin tapes with higher volume fractions of fiber to reduce the amount of debulking that currently must be done. A lower CTE backing paper is desired to make the manufacturing of thin ply prepregs easier.

It is not believed that the PC resins, when supplied to the prepreg manufacturers and parts fabricators, are hazardous. The dangerous part of the process is cyanation which is performed during resin manufacture. Whether facilities currently performing the cyanation process will encounter future environmental concerns is not yet known. At this time the cyanation step for the Dow resin is performed by Lonza in Europe, which is generally thought to have environmental regulations at least as strict as the United States.

### C. CONCLUSIONS AND RECOMMENDATIONS

The spacecraft community is clearly interested in the high-performance graphite/polycyanate composites. And, just as clearly, there are some issues that must be resolved before these materials will see more widespread use. These issues can be divided into two categories: materials/processing and data/analyses. Specific recommendations in these areas are outlined below for consideration by the workshop participants.

### 1. Materials and Processing

Recommendations in this category are related to material development and processing experience.

(a) "Product creep" resulting from changing material formulations is a continual problem in the advanced materials business. It is also apparent that the time and funding available for material qualification is decreasing. Therefore, these materials must be standardized to the extent possible to increase their acceptance by designers. It may be necessary to quantify the benefit of these minute changes in material composition to justify their use by designers. In fact, it may be impossible to generate a satisfactory database unless the

- materials become somewhat fixed over time. This is an issue that can only be resolved by industry.
- (b) Recommended topics to be addressed for the resins include development of low-temperature curing systems with high Tg's; investigation of Tg as a function of post-cure treatments; determination of additive-rheology-tack relationships; development of additives for resin toughening; and matrix studies of microcracking resistance.
- (c) In terms of prepreg development, lower resin contents in ultra-thin tapes should be considered for spacecraft applications.
- (d) Coatings for environmental protection and processes to apply them also need to be developed for spacecraft applications.
- (e) Structural bonding processes require additional development. In particular, new adhesives with appropriate fillers and additives are desired. Designers do not want to give up the low outgassing attributes of the PCs by requiring high outgassing adhesive formulations for joining.
- (f) General processing goals to be addressed include development of low cost in elligent processing approaches, nonautoclave curing methods, quality control and NDE procedures, and machining methods. Development of a process specification will also lead to increased user acceptance.

### 2. Data and Analyses

Additional data is needed, particularly on the effects of space environments on properties. Such that can probably be best obtained by companies (end users, materials suppliers, and fabricators) working together, perhaps in a "round robin" format using agreed upon materials, designs, and test procedures. Commonality of test techniques will be a key factor in making such an effort successful.

- (a) Needed mechanical/physical property data include dimensional stability/creep, fatigue life, fracture toughness, damage tolerance, and post-cure effects.
- (b) Environment-related data, including their effects on mechanical and physical properties are required on: low (cryogenic) temperature mechanical properties; thermal cycling and microcracking resistance; moisture and thermal cycling effects on dimensional stability; EMI/ESD; atomic oxygen erosion; radiation and other LEO effects; and contamination from outgassing.
- (c) Data on the adherence of protective coatings during processing and mission operations is required.
- (d) Joint tests and subsystem level tests of structural component e also required for increased designer acceptance.

Analytical design tools, suggested below, require development as well. These will no doubt occur over time as advanced composites are increasingly used in real applications.

- (a) Spacecraft design approaches need to be developed in light of smart structures and/or autonomous control technologies. Other tools to be developed may include optimization codes for minimum spacecraft weight; nonstructural design standards for composites; analytical codes for multicomponent anisotropic material systems; and determination of empirical design relationships.
- (b) Flight experiments appear to be a very key step in creating designer acceptance of an advanced material. Current budget environments within the government as well as its contractors may not permit such experiments to be done. Options include development of acceptable ground tests or establishment of an expert review board to determine if flight testing is needed for material qualification.

### 3. Recommendations for BMDO

As mentioned above, the current budget environment does not permit the government to support all the activities described above. Based on discussions throughout the workshop it seems clear that the major issue of concern to the designers was lack of a statistical/design database on graphite/polycyanate composites. Development of a data handbook via government coordination of the existing information may be one way to promote pooling/sharing of available data and provide assessments of data quality so contractors can build on past work. This appears to be critical to increased designer acceptance. It will also lead to more effective use of corporate research dollars by avoiding duplication of effort.

### APPENDIX A AGENDA AND ATTENDERS

### Workshop on Graphite-Reinforced Polycyanate Composites

### June 16, 1993

### Final Agenda

	Topic	Speaker	
7:45 am	Begin check-in, coffee, etc.		
8:05	Welcome	J.M. Sater, IDA	
8:10	Introduction	LtCol M. Obal, BMDO	
8:30	Why Polycyanate Composites?	J.M. Sater	
I. Spacecraft Designers			
8:50	The Aerospace Perspective	M.Aswani, Aerospace	
9:10	TRW Perspective	L. Rosales, TRW	
9:30	OS Perspective	T. Dragone, Orbital Sciences	
9:50	Ball Perspective	M. Dean, Ball Space Systems	
10:10	Break		
10:30	Lockheed Perspective	G. Ritchie, Lockheed	
10:50	MM Perspective	R. Hansen, Martin Marietta	
11:10	Loral Perspective	J. Cooney, Loral	
11:30	Rockwell Perspective	W. Harvey, Rockwell	
11:50	Lunch		
12:40	Hughes Perspective	P. Burns, Hughes	
II. Prepreg Material Suppliers			
1:00	Dow Update	T. Benson Tolle, WL	
1:10	Hexcel Perspective	B. Gilmore, Hexcel	
1:30	ICI Fiberite Perspective	D. Powell, ICI Fiberite	
1:50	YLA Perspective	G. Patz, YLA, Inc.	

### III. Parts Fabricators

2:10	GBI Structures, etc.	C. Barker, Sparta
2:30	Break	
2:50	Clementine Panels, etc.	J. Koury, Phillips Lab
3:10	SAWAFE Panels, etc.	S. Rawal, Martin Marietta
3:30	Clementine Panels, etc.	G. Krumweide, Composite Optics
3:50	ACTEX, etc.	R. Lewis, TRW
4:10	All-Composite Spacecraft, etc.	H. Dursch, Boeing
4:30	GSTS Structures, etc.	J. Tracy, McDonnell Douglas
4:50	Break	
5:10	Discussion and Closing Remarks	

### Workshop on Graphite-Reinforced Polycyanate Composites Names and Addresses

### I. Spacecraft Designers

Mohan Aswani
The Aerospace Corporation
M4/920, P.O. Box 92957
Los Angeles, CA 90009

310-336-4440 fax 310-336-1474

Tom Dragone Orbital Sciences 21700 Atlantic Blvd. Dulles, VA 20166 703-406-5413 or 802-8107 fax 703-802-8255

Lou Rosales TRW One Space Park 01/2210 Redondo Beach, CA 90278 310-813-9201 fax 310-812-8768

Mike Dean
Ball Space Systems Division
MS CEC
10 Longs Peak Drive
Broomfield, CO 80021

303-460-3748 fax 303-460-3537

William A. Harvey Mail Code SX08 Rockwell International 2800 Westminster Blvd. P.O. Box 3089 Seal Beach, CA 90740-2089 310-797-4870 fax 310-797-4796

John Cooney Space Systems/Loral MS G97 3825 Fabian Way Palo Alto, CA 94303 415-852-4703 fax 415-852-4267

Bob Hansen Martin Marietta Aerospace Box 179, MS/B3085 Denver, CO 80201 303-971-9390 fax 303-971-9141

Gordon Ritchie Lockheed Dept. 77-20/551 P.O. Box 3504 Sunnyvale, CA 94088-3504 408-743-1588 fax 408-756-2188 Pam Burns
Hughes Space & Communications Group
Bldg S52, MS K311
P.O. Box 92919
Los Angeles, CA 90009

310-364-8462 fax 310-416-6971

### II. Material Suppliers

Gary Patz YLA, Incorporated 2970 Bay Vista Court Benicia, CA 94510 707-747-2750 fax 707-747-2754

Dave Powell ICI Fiberite Composite Materials 2055 East Technology Circle Tempe, AZ 85282 602-730-2213 fax 602-

Brian Gilmore Hexcel Advanced Composites 5794 West Las Positas Blvd. Pleasanton, CA 94588 510-847-9500, ext 4428 fax 510-734-8013

Scott Unger Bryte Technologies 1840 Milpitas, CA 95035 (This is a bonus package.) 408-946-6477 fax 408-946-9388

### III. Composite Parts Fabricators

Christine Barker SPARTA 9455 Towne Centre Drive San Diego, CA 92121-1964 619-455-1650 fax 619-455-1698

Jim Koury Phillips Laboratory OLAC PL/VTSC Edwards AFB, CA 93523 805-275-5646 fax 805-275-5144

Suraj Rawal Martin Marietta Aerospace Box 179, MS/B3085 Denver, CO 80201 303-971-9378 fax 303-971-9141

Gary Krumweide Composite Optics, Inc. 9617 Distribution Avenue San Diego, CA 92121 619-566-5591 fax 619-566-7204 Rich Lewis
TRW Electronics & Space Division
One Space Park
MS 01/2220
Redondo Beach, CA 90278

310-813-9227 fax 310-

Harry Dursch Beoing Defense and Space Group P.O. Box 3999 MS 82-97 Seattle, WA 98124-2499 206-773-0527 fax 206-773-4946

John Tracy McDonnell Douglas 5301 Bolsa Avenue MS A3/13-3 Huntington Beach, CA 92647 714-896-5169 fax 714-896-6930 email - TRACY@apt.MDC.COM

Also: LtCol Mike Obal (BMDO) John Stubstad (BMDO) Mike Rigdon (IDA) Janet M. Sater (IDA) Bob Acree (PL) Tia Benson Tolle (WL) Jan Garrison (WL) Jim McCoy (WL) Bill Cameron (WL) John Dignam (ARL) Roger Crowson (SSDC) Joan Funk (NASA-Langley) Ainslie Young (LANL) Nick Davinic (NRL) William Braun (NRL) Al Bertram (NSWC)

### APPENDIX B INTRODUCTORY PRESENTATIONS

# The Materials and Structures Program

# Workshop on Graphite-Reinforced Polycyanate Composites

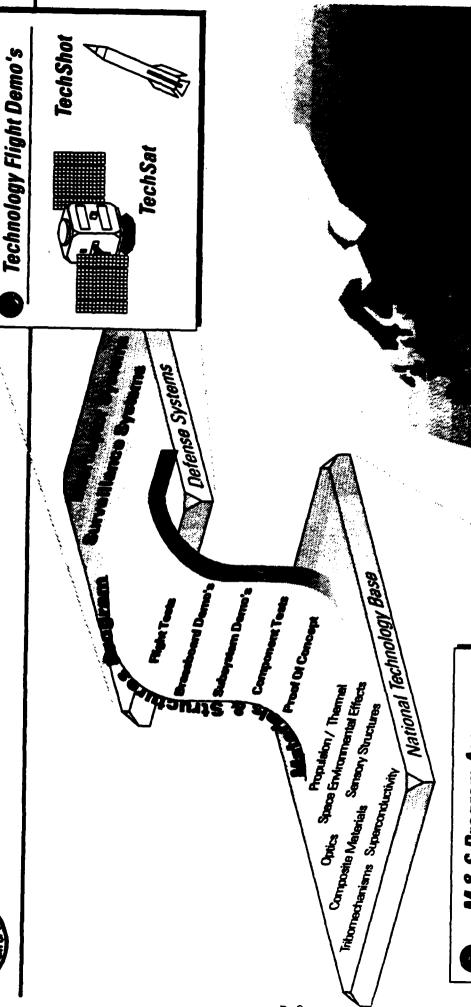


LtCol Michael Obal, Program Manager

Dr. John Stubstad, Deputy Program Manager

Institute for Defense Analyses Alexandria, VA June 16, 1993

# M&S Program Evolution

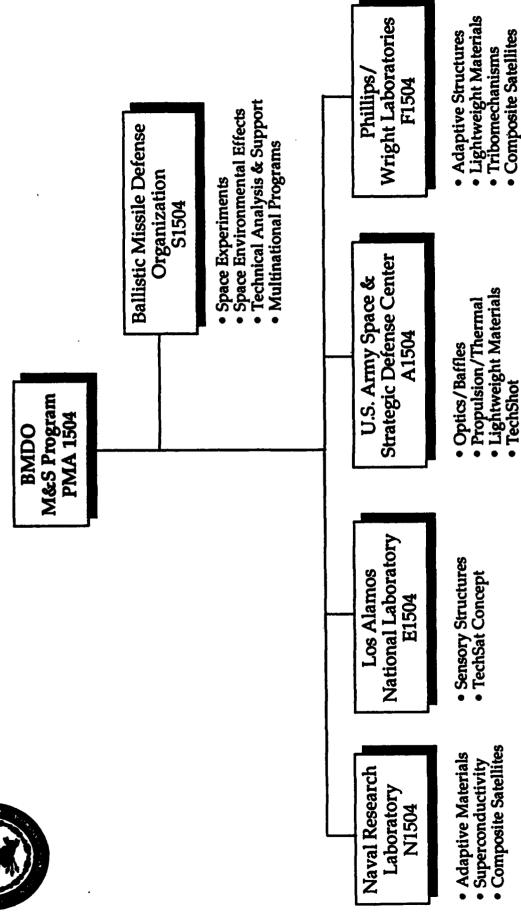


M & S Program Areas

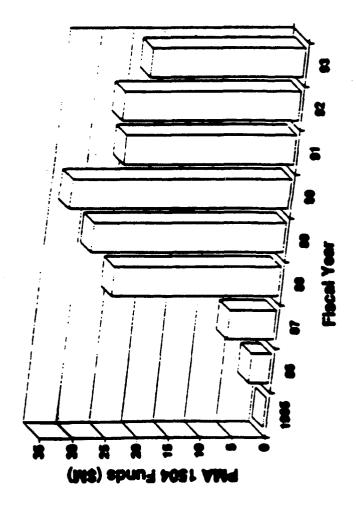
- Adaptive Structures
- Space Evironmental Effects
- Lightweight Structural Materials Optical Materials
- **Tribomechanisms**
- Propulsion/Thermal Superconductivity

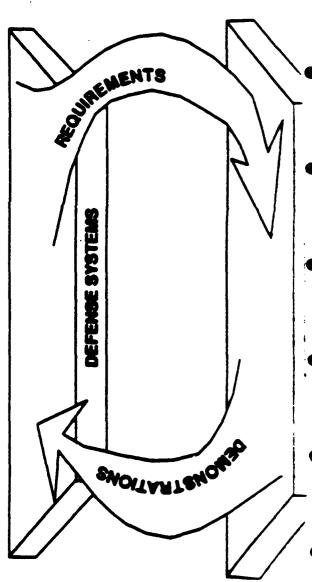


# THE M&S ORGANIZATION



TechŠat Concept





## Critical Technologies PMA 1504/1703 Contribute to BMDO

- Jitter Control
- Light Weight Structures
- Space Environmental Effects
- Low Cost Manufacturing
- Sensor System Performance





# PMA 1504/1703 Charter Implementation

- Reduce Risk
- Enhance Capabilities
- Increase Affordability

Issue Identification

Issue Resolution



**Assessments** SPO/Agent

SDIO PE Managel Interchanges



Test

On-orbit

Experiments

Sounding Rocket Test Bada

Direct Involvement

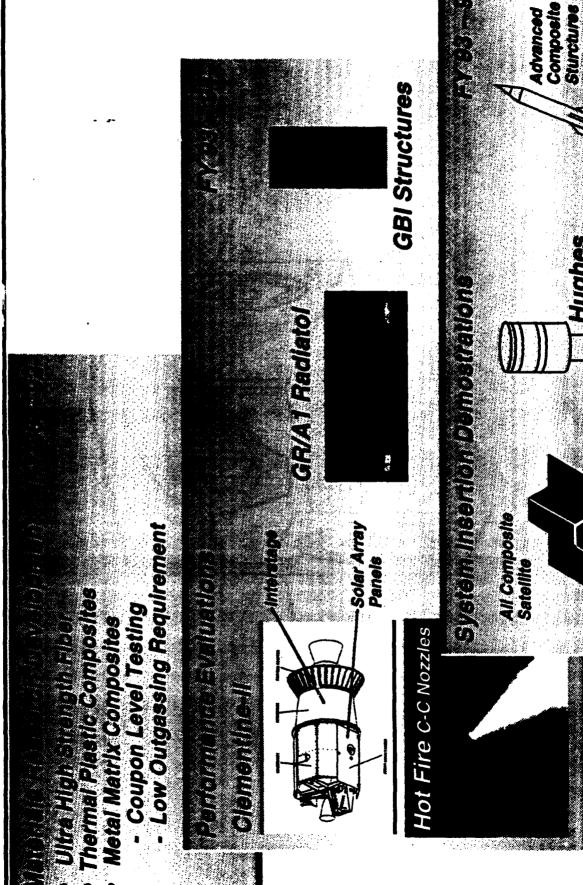
SPO/Contractor



**GDI**.X

## Materials & Structures PMA 1504

# **Light Weight Structures**





# Low Cost Manufacturing









D-2 AEROSHELI C-C Nozzle FY 97 = 93

Array Structure

Sogrid Solar





**Alternative** 

Automated Methods BP Interstage Structural Design



High Temp. Composite Structure



FY 93 - 96

TechSho





**B-8** 



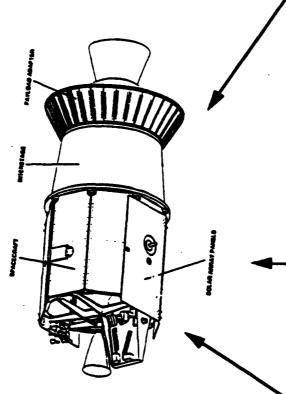


### - Clementine -

 Gr/Polycyanate solar array substrate

· Gr/Epoxy interstage laminate structure

- Isogrid Structure
- IM7 fiber
- Bryte BTCY-1A cyanate ester resin
- · Est. 11 lb wt. savings over Altıminum

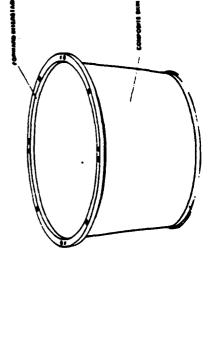


Ciba Geigy 6376
 epoxy resin

T650 fiber

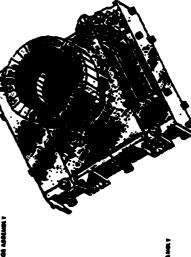
• Est. 15 lb wt. savings over Aluminum

Interstage Structure



Solar Array Assembly







## **Advanced Composites**

### **MILITARY APPLICATION**

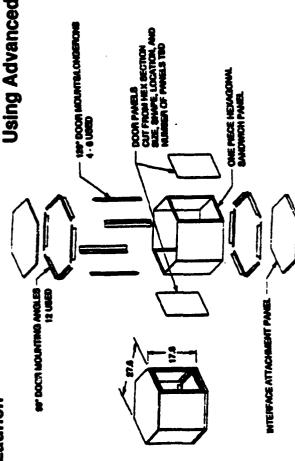
Low Cost Light Weight Producible Satellite Design

### CIVIL APPLICATIONS

Satellite Design for Low Production/Launch

### **TECHNOLOGY BENEFIT**

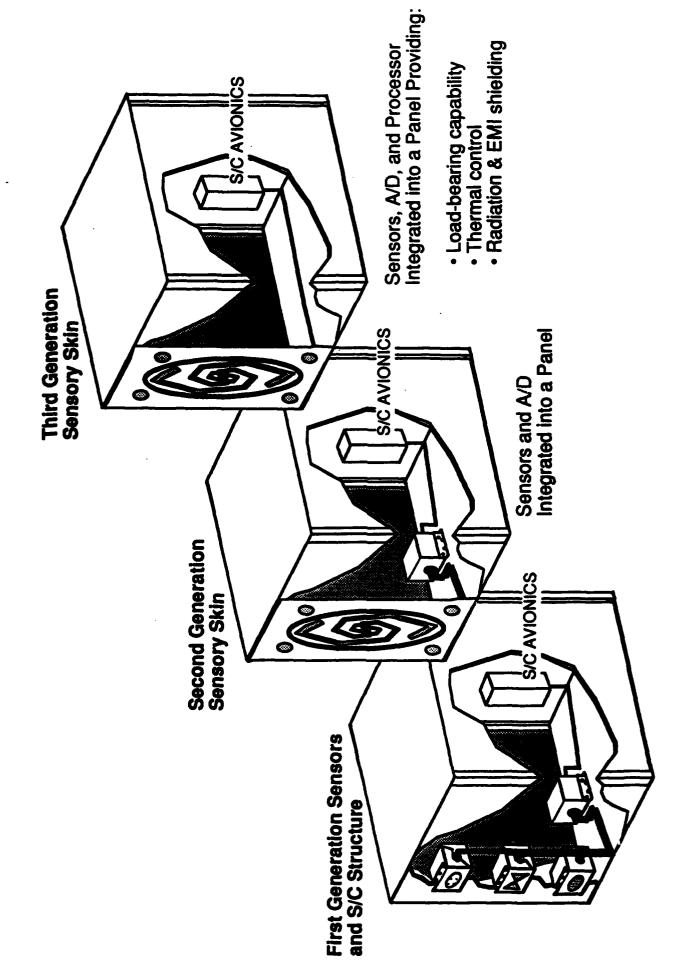
- Launch/Space Load Evaluation of Ultra-Stiff Fiber System Composite Structure
  - Provide Outgassing and Dimensional Stability Data
- Provide Cost/Schedule Data for Low-Cost
  - Manufacturing Techniques
    Demonstrate Bus Integration Methods
    Using Advanced Composite Structures



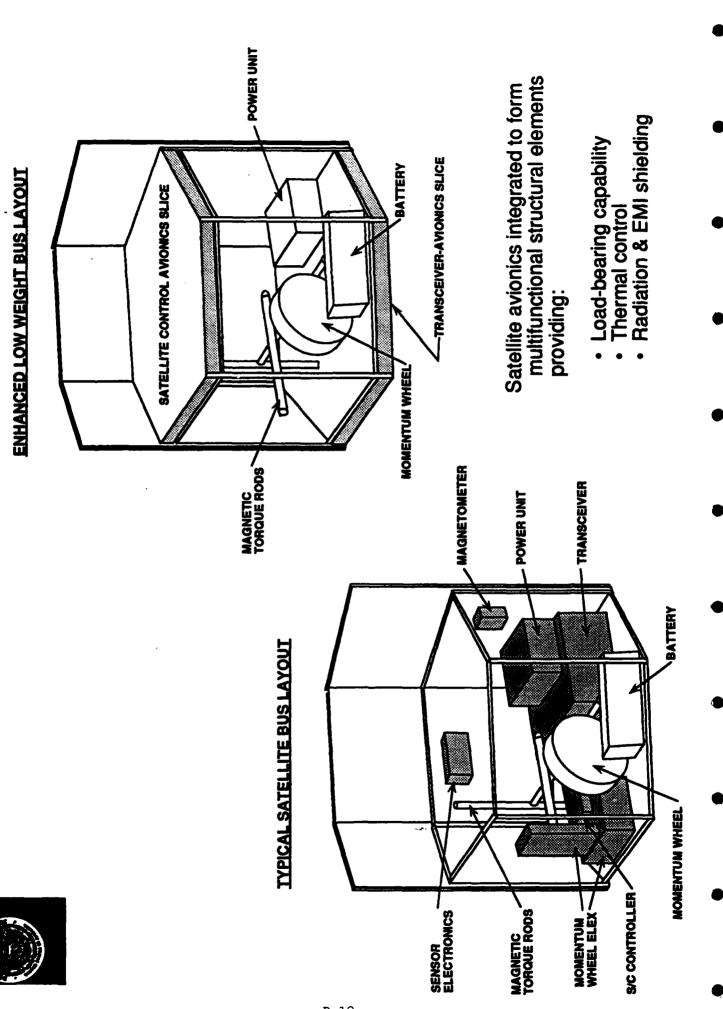
Source: Boeing/USAF Phillips Laboratory

WJSA.MDR.4.29.93.14

# SENSORY STRUCTURES TECHNOLOGY



# **MULTIFUNCTIONAL INTEGRATED STRUCTURES TECHNOLOGY**



# General Workshop Objectives

 Increase communication among designers, material suppliers, and parts manufacturers

application, processing, and manufacture of graphite-- Identify technical issues and limitations associated with reinforced polycyanate composites

# Why Polycyanate Composites?

Janet M. Sater

Graphite-Reinforced Polycyanate Composites Workshop on

Institute for Defense Analyses Alexandria, VA

June 16, 1993

## SDIO/BMDO Programs

### INITIAL FOCUS

### CURRENT FOCUS

### SYSTEMS Interceptors/missiles

Non-acquisition 1000s (SBI, ERIS, E<sup>2</sup>I)

Acquisition 100s (BP, GBI, TMD, THAAD)

Ground/space structures

Very large (NPB, SBL, SBR, BSTS, SSTS, GSTS, GBR)

Moderate to small (BE, GBR)

## REQUIREMENTS

Weight Strength Stiffness Damping, vibration isolation Thermal fatigue

Low High High Very rapid response Very long life Zero/Near-zero Very high Very long times on orbit, extreme threat Low but more performance-driven

environment resistance

Dimensional stability

Natural and threat

Thermal expansion

resistance

performance

High High Rapid response Long life Moderate High Moderate times on orbit, low/no threat Low, ~equal with

**ATTRIBUTES** 

# M&S Response to Program Needs

### Initial Focus

**Current Focus** 

Candidate material evaluation Coupon, tube testing C/C

MMCs Gr/Al, Gr/Mg, SiC/Al

**PMCs** 

Gr/TS, Gr/TP

Systems insertion demonstrations

> components and structures Fabrication of demo

## M&S Response - Evaluation

<u>(၂</u>

Threat survivability

**MMCs** 

Gr/Mg, Gr/Al

High specific strength, modulus

Near-zero CTE

No outgassing

Natural & threat environmental resistance

SiC/AI

Close CTE match for mirrors Relatively high modulus Producibility

# M&S Response - Evaluation (contd)

PMCs Gr/Epoxy Availability

Improved microcracking resistance Higher T use Producibility High specific strength, modulus Processability, reprocessability Improved toughness/damping -ow outgassing **Gr/Thermoplastics** ow CTE

# M&S Response - Fabrication Experience

Coupons

Tubes, struts, panels, frames

Joints

Seeker structures

Interceptor structures

**Bus structures** 

Trusses

Adaptive structures

## M&S Response - Issues

Reality of trade studies

Material quality Uniformity Reproducibility Residual stresses

Material availability Fiber Thin plies Material/processing cost Equipment

Joining

Where did polycyanate composites come from?

T<sub>g</sub> ≥ molten solder T (~220-270°C) Low dielectric properties Glass, aramid fibers Good peel strength Circuit Board Industry

Good translation of mechanical properties using Fabrication via a number of processes without Reduced moisture absorption, outgassing Addition of toughening agents changes in performance Where have they gone since then? Tougher than epoxies, BMI Lower dielectric properties  $190^{\circ}\text{C} \le T_g \le 290^{\circ}\text{C}$ various fibers

## Known Potential Applications

### Circuit boards

## Automotive components

Torque-arresting spring for automatic transmission Coupling plate for a filament-wound drive shaft Brake-caliper, disk-cooling ducts ('92 Indy car) Monocoque chassis

## Missile nose cones

Radomes, high-gain antennas

Primary, secondary structure for aircraft Stealth aircraft

Stealth aircraft High Speed Civil Transport European Fighter Aircraft

# Known Potential Applications (contd)

## Satellite structures

Tubes, struts, panels Optical bench, IR mírrors Filters, wave guides, feed guides, and horns Phased array antennas

### Liquid hydrogen storage tanks National Aerospace Plane

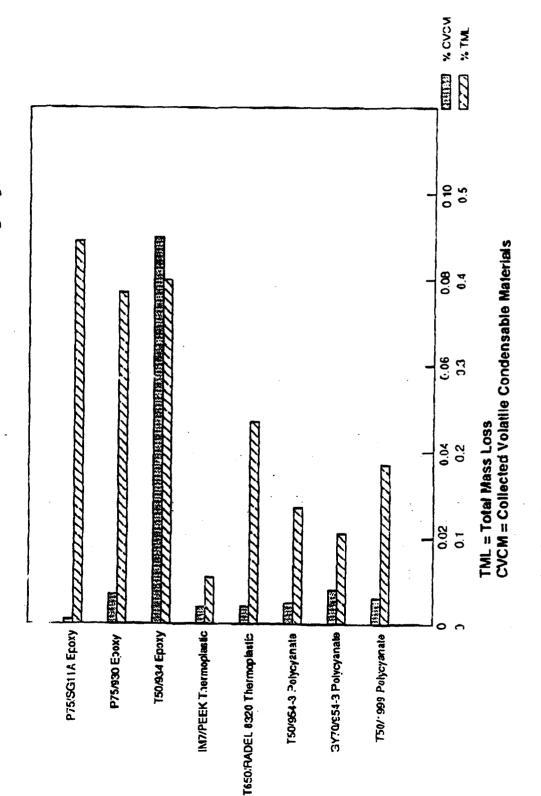
Cryogenic, radiation-resistant components Superconducting Supercollider

### Others





## Epoxies, Thermoplastics, and Polycyanates **Outgassing Properties of**



## Potential Benefits

Industry/contractor interest Processing facilities Financial support

Epoxy-like processing Higher T<sub>g</sub> Good processability

Resistance to microcracking (thermal cycling) Low cure shrinkage (<1%) Inherent toughness

Low moisture pick-up, outgassing

Near-zero thermal expansion coefficient

### Topics - Designers

secondary structure) including properties, qualification Necessary requirements/data for the application of advanced materials to real system (primary and tests, etc.

Other issues that need to be addressed for such application Specific fiber/matrix combinations that may be of interest

## Topics - Material Suppliers

- Information needed from designers to insure adequate material development
- Current availability of data including material options (fiber (quantities produced), density, physical and mechanical architectures, matrix options) and availability/maturity microcracking and thermal cycling, natural space environment survivability (e.g., atomic oxygen, properties, contamination and outgassing, radiation)
- Manufacturability/producibility and joining issues
- Estimated current and projected costs
- Other potential risks

# Topics - Composite Parts Fabricators

Current experience with graphite-reinforced polycyanate composites, particularly material uniformity and processing-related issues

Results/data from any additional testing (including data already identified)

especially those with which you have had problems Actual components/parts that have been fabricated,

#### APPENDIX C DESIGNER PERSPECTIVES

GRPC 6/16/93

## GRAPHITE-REINFORCED POLYCYANATE COMPOSITE WORKSHOP

# Material Selection and Qualification

Mohan Aswani Structural Technology Department

16 June 1993

### OVERVIEW

- Material Selection
- What to consider
- What to avoid
- Material and component qualification
- Features
- **Model Plan**
- Lessons learned example
- Traits of successful application



# MATERIAL SELECTION - WHAT TO CONSIDER

System requirements flow down

Subsystem / Component weight

Stiffness / Strength, Thermal conductivity

Dimensional stability

Interface with other subsystems

Storage / Operational environment

Candidate materials

Physical and thermo-mechanical properties

**Processability** 

Application related experience

Supply source and cost

Trade studies

Selection criteria

Design flexibility

Credible models

**Critical parameters** 

Schedule and cost

# MATERIAL SELECTION - WHAT TO AVOID

- Requirements allocation based on
- Inadequate performance margins
- Unrealistic weight allocation
- Unsubstantiated promises of high performance
- Candidate materials chosen based on
- Very limited properties data
- Little or no experience
- Unreliable supply source
- Uncertain cost and schedule impact
- Trade Studies Performed
- With pre-determined outcome
- Without credible analyses
- Without subsystem interaction considerations



# MATERIAL & COMPONENTS QUALIFICATION

#### **FEATURES**

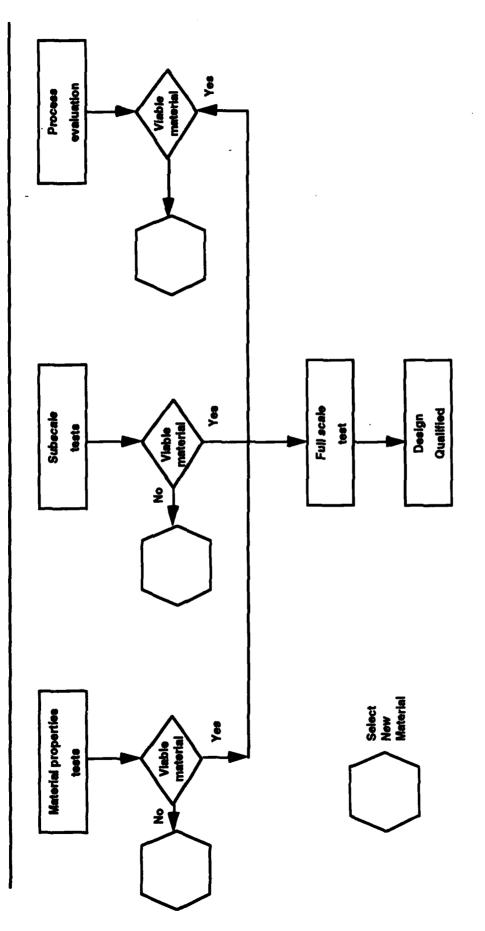
- No shortcuts driven by program schedule
- Thorough system impact evaluation
- Balanced program based on analyses and tests

C-5

- Quantification of material properties and processes variability
- Proven test and inspection procedures



# MATERIAL QUALIFICATION PLAN





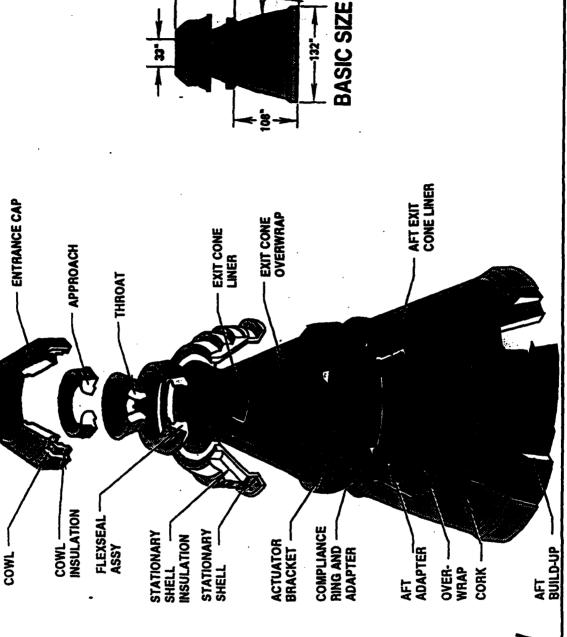
# LESSONS LEARNED EXAMPLE

Alternate material for solid rocket motor nozzles

Replacement of rayon-based carbon phenolic by Pan-based phenolic nozzle components



## ROCKET MOTOR NOZZLE



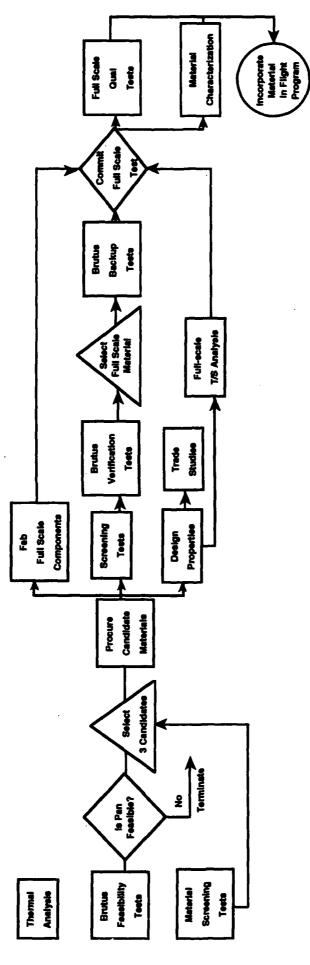
C-8

EXPLODED VIEW

THE AEROSPACE CORPORATION

## LESSONS LEARNED

### Approach A





## LESSONS LEARNED

- Results of Approach A
- Excessive defects (porosity / delaminations) in full scale components due to lack of process control
- Structural test failure full scale properties different from coupon
- Reduced thermal / structural margins required redesign of some components
- NDI techniques not directly applicable new standards required
- Wastage of full scale components due to unacceptable material deficiencies

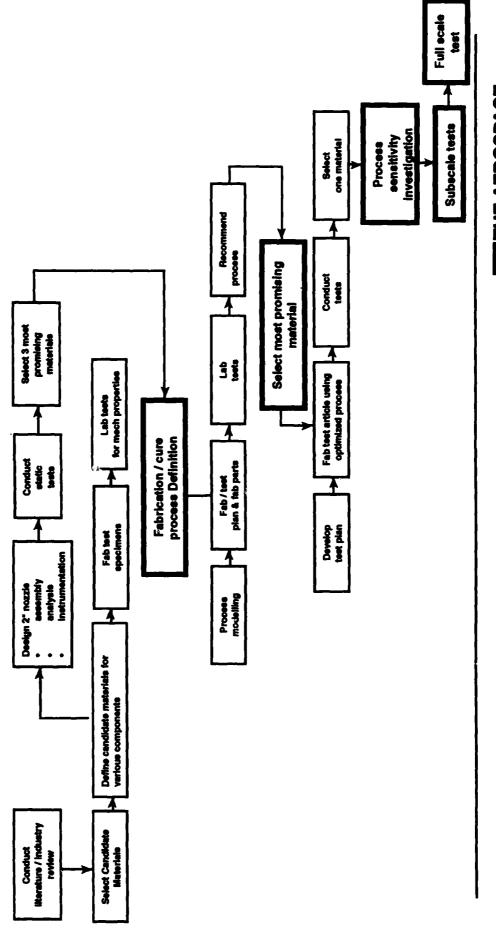
Pan-nozzle program discontinued: cost several million dollars



## LESSONS LEARNED

Evaluate candidate materials

Approach B



THE AEROSPACE CORPORATION

# TRAITS OF SUCCESSFUL APPLICATION

- Adequate material property database
- Relevant experience with similar materials and applications
- Good system engineering approach and balanced allocation of requirements
- Credible material trade studies
- Acceptable design and analysis practices including attention to joint designs and manufacturing limitations
- Well planned test program and analysis / test correlation
- Proven processing specifications and demonstrated producibility
- Cost effective





# USE OF POLYCYANATE RESIN COMPOSITES IN SPACECRAFT DESIGN

DR. LOUIS A. ROSALES

WORKSHOP ON GRAPHITE-REINFORCED POLYCYANATE COMPOSITES

16 JUNE 1993 ALEXANDRIA, VIRGINIA



# SPACECRAFT DESIGN MATERIAL ISSUES

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### MATERIAL REQUIREMENT

### SENSOR OR INSTRUMENT PERFORMANCE

### DIMENSIONAL STABILITY

## LOW MOISTURE ABSORPTION/CME

### THERMAL MANAGEMENT

#### LOW WEIGHT

**USE HIGH PERFORMANCE COMPOSITES** 



### TRW DEVELOPING LINE OF ADVANCED BUSES FOR **NEAR-TERM AND FUTURE SPACECRAFT**

- WIDE RANGE OF SPACECRAFT SIZES <1000 LB TO >10,000 LB 0
- **DESIGN-TO-MANUFACTURING CONCEPT STANDARDIZATION** 0
- 0 "TAILORABLE" STRUCTURE MODULARIZED
- 0 CONTINUOUS TECHNOLOGY INSERTION
- 0 ALL COMPOSITE STRUCTURE
- FIRST PROTOTYPE FABRICATED USING EPOXY GFRP
- SECOND GENERATION BASED ON POLYCYANATES

### Advanced Bus 600 (AB600)

operational missions as communications, science, surveillance and remote sensing. The TRW Advanced Bus 600 (AB600) is a satellite designed to host large payloads ightweight, low-cost, multi-mission typically flown for such long-term

llexibly designed to accommodate a range up to 10 years. Little change is needed to meet mission-specific needs. Technology of payloads weighing up to 1,000 pounds. up to 800 Watts and on-orbit lifetimes of It supports payload power requirements The AB600 is compact, modular and insertion is readily accommodated hrough standardized interfaces.

propellant for on-orbit stationkeeping and an avionics and propulsion module, which can provide over 200 pounds of hydrazine payloads to easily "bolt-on" to the AB600 other functions. An additional module is core configuration. The core consists of electrical interfaces allow customer available to accommodate mission-Standard mechanical, thermal and specific equipment.



specific Module

#### Configuration

racking, Telemetry &

- 6-sided, 38-inch diameter
  - 34 inches high
    - 380-500 lbs (dry)

#### **Attitude Control**

- Knowledge to <0.10\* 3-axis stabilized
  - Control to <0.10

Pegasus **Faurus** Delta II Atlas II Titan II

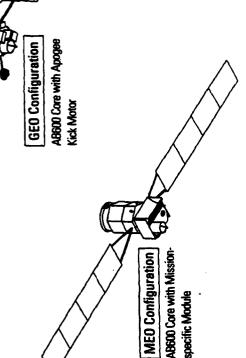
#### **Electrical Power**

- 45 amp-hr NiH2 or NiCd battery Silicon or GaAs solar arrays
  - Sized to requirements
- Articulated solar arrays

### Payload Accommodations

- Lifetimes up to 10 years
  - Up to 1,000 lbs
- Up to 800 W (peak) Orbits LEO, MEO, HEO, GEO

#### S-band (SGLS, STDN, others) Command /Data Handling Downlink up to 5 Mbps Secure encrypted links Mass memory options Launch Vehicle Jplink 2 kbps



LEO Configuration

AB600 Core

**Multi-mission Capabilities** 

Printed in the USA SEEG Graphy Communication FD 7220 + CP + 3M + 4/53 945



# ULTRALIGHTWEIGHT DIMENSIONALLY STABLE COMPOSITES

SPACECRAFT REQUIRE LARGE DIMENSIONALLY STABLE COMPONENTS

- USUALLY DEPLOYABLE
- STABLE UNDER ATMOSPHERIC MOISTURE/VACUUM DRY-OUT
- STABLE WITH TEMPERATURE CHANGES
- STABLE WITH TEMPERATURE CYCLING
- STABLE UNDER VIBRATIONAL OR TRANSIENT LOADING
- MAY REQUIRE PASSIVE OR ACTIVE DAMPING
- TYPICAL COMPONENTS INCLUDE ANTENNAS, METERING TRUSSES AND OPTICAL BENCHES



# **ULTRALIGHTWEIGHT DIMENSIONALLY STABLE COMPOSITES**

ADVANCED COMPOSITE MATERIALS ARE USED FOR COMPONENTS

- HIGH STIFFNESS GRAPHITE FIBERS (UP TO 130 MSI)
- LOW MOISTURE ABSORBING, MICROCRACKING RESISTANT POLYMERIC MATRIX MATERIALS
- **POLYCYANATES**
- MODIFIED EPOXIES
- CO-POLYMERS
- NEED ULTRA-THIN PLIES TO ALLOW 0 CTE LAMINATES
- 0.001 INCH PER PLY OR LESS

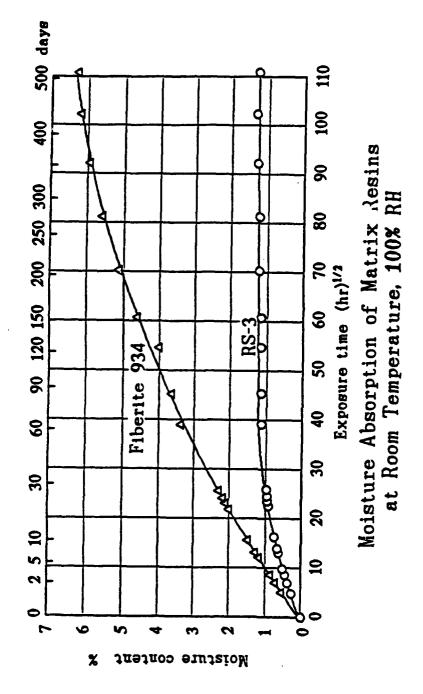


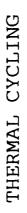
Materials Technology & Engineering Spacecraft Technology Division

# Polycyanate Properties Versus Epoxies

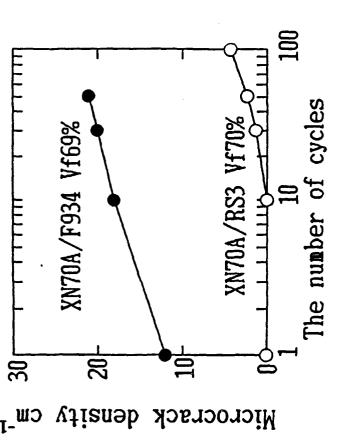
Neet Beein	Typical Values (350°F Cure)	s (350°F Cure)	Affected Cteresties
Properties	Polycyanates	Epoxies	Parameter
Density (g/cm)	1.19	1.25 to 1.3	Weight
Tg (°F)	390 to 650	360 to 450	Operating temperature
Flexural strength (ksi)	12.5	10	Matrix strength
Flexural modulus (MSI)	0.40	09:0	Matrix stiffness
CTE in/in°F (-212°F, +300°F)	26.0	27.0	Thermal distortion
TML% \ per	0.1 to 0.3	0.3 to 1.5	Contamination
CVCM% / ASTM 595E	0 to 0.01	0.01 to 0.8	Contamination
Moisture absorption (%)	0.6 to 2.5	4 to 7	Moisture stability contamination
Dielectric properties Dk Df	2.5 to 3.1 ~0.007	3.5 to 4.0 ~0.04	RF performance RF performance

YLA DATA









Results of Microcracking Determination Test



## **EPOXIES VS. POLYCYANATES**

### MANUFACTURABILITY

- **CURE AT SAME TEMPERATURES**
- CAN USE EXISTING TOOLING/OVENS/AUTOCLAVES/ETC.
- SOME SLIGHT DIFFERENCES IN TACK
- POLYCYANATES HAVE BETTER "OUT-TIME"
- BOTH CAN BE COMBINED WITH GRAPHITE OR KEVLAR FIBERS

#### DATA BASE

- **EPOXIES HAVE WELL ESTABLISHED DATA BASES**
- **EPOXIES ARE FLIGHT PROVEN**
- POLYCYANATES HAVE LIMITED DATA BASE
- NO TRW FLIGHT DATA





Approximately four years of use at TRW in specific applications

IR&D or technology programs which demonstrated a specific application, usually did not develop comprehensive material data Excellent experience in processing into a variety of structural shape and forms

- Tubes: both thin-walled, dimensionally stable and structural, including smart
- Membranes: ultra-thin, dimensionally stable, both pure laminates or sandwich construction
- Honeycomb: ultra-light, all GFRP

Some material data was generated during these programs. Several different graphite fibers were used TRW has also obtained data from vendors and other spacecraft companies on specific fiber/resin composites Our experience has been in developing selected components rather than in material development data



# CURRENT TRW POLYCYANATE DEVELOPMENT ACTIVITIES

- 0 DESIGN DATA BASE DEVELOPMENT
- REQUIRED TO DESIGN FOR WEIGHT EFFICIENCY
- REQUIRED TO DETERMINE MARGINS OF SAFETY
- "B"-BASIS DESIGN ALLOWABLE
- 90% OF SAMPLED MATERIAL EXCEEDS VALUE WITH A 95% CONFIDENCE LEVEL
- TRW REQUIRES SAMPLING FROM 3 LOTS OF MATERIAL
- 0 PROCESSING DATA DEVELOPMENT
- SPACECRAFT SUBELEMENT DESIGN/FABRICATION/TEST
- PERFORMED AS PART OF TRW ADVANCED BUS INITIATIVE



## "B"-BASIS DESIGN DATA DEVELOPMENT

**BOTH PAN AND PITCH BASED FIBERS** 

TAPE AND CLOTH FORMS

RANGE OF STRENGTHS AND MODULI

TRW WORKING WITH MATERIAL SUPPLIERS TO DEVELOP DATA

FIBER SUPPLIERS

RESIN SUPPLIERS

TRW PERFORMING IRAD TASKS FOR FULL OR PARTIAL DATA DEVELOPMENT

TRW PERFORMING "SPOT CHECK" OF DATA BASE DEVELOPED BY **OTHER COMPANIES (DATA SHARE)** 

TRW PURSUING FUNDING TO ADD FIBER/RESIN SYSTEMS

## OSC Perspective on Advanced Composite Materials

Orbital Sciences Corporation

Dr. Tom Dragona Machanical Engineer OSC / Space Systems Division 21700 Atlantic Boulevard Dulles, Virginia 20166 (703) 406-5000

## **OSC Design Philosophy**



- Take Advantage of State-of the-Art in all Appropriate Technologies (even if extensive heritage doesn't exist)
- Rapid Shift from Design through Development to Production
- Early Prototyping
- Extensive Engineering Testing
- Focus on Reducing Cost to Orbit
- Use "Off the Shelf" Materials where Appropriate
- Use Advanced Materials where Specific Performance Advantages are Obvious

⇒OSC is Willing to Take Calculated Risks in Using Materials that are not Fully Characterized

## OSC Design Philosophy in Practice

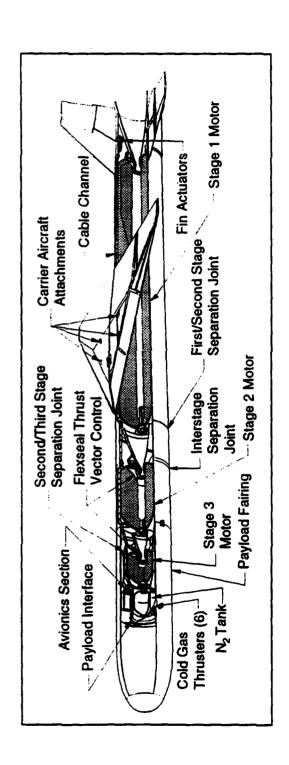


#### Pegasus

- All-Composite Structure (Motor Case, Wing, Fins, Nozzles)
- Advanced Propulsion, Avionics and TVC Technologies
- PegaStar Small Satellite Bus (APEX, SeaStar)
- "Off the Shelf" Materials
- Modular Design Approach
- Integrated Bus Reduces Complexity and Increases Payload Fraction
- MicroLab Small Satellite Bus (ORBCOMM, LIS/GPSMet)
- Sophisticated Metallic Alloys and Composites
- Rapid Turnaround: 6 Months from Contract to Launch

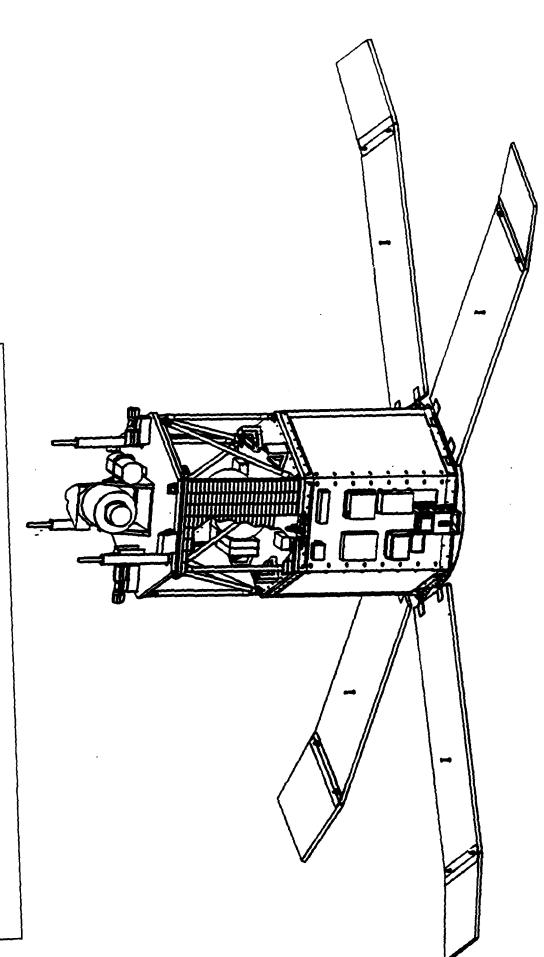
### Pegasus Air Launched Space Booster Cut-Away Schematic





# PegaStar Integrated Satellite Bus

Orbital Sciences Corporation



OSC / Space Systems Division 21700 Atlantic Boulevard Dulles, Virginia 20166 (703) 406-5000

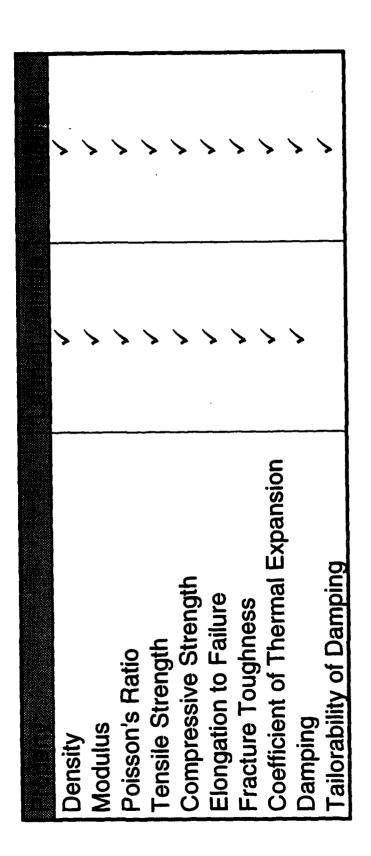
#### Sciences Corporation

### **Design Requirements**

- Launch Vehicle Requirements Driven by:
- Short Life Cycle (<15 min)
- Uniformity / Manufacturing Concerns
- Satellite Requirements Driven by:
- Long Life Cycle (>≈5 Years)
- Unique Mission Constraints

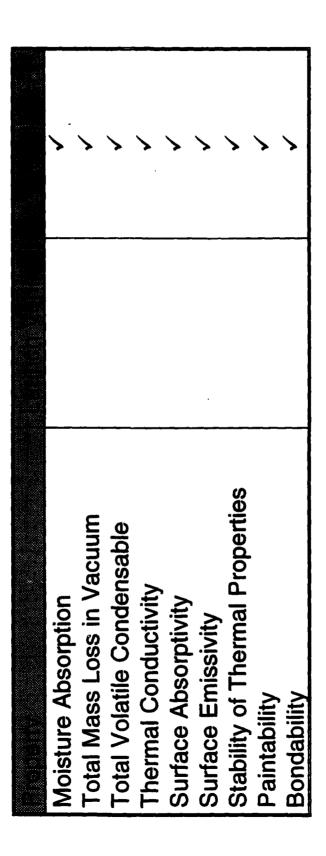
## Design Requirements Mechanical / Physical Properties





### Sciencies Corporation

### Design Requirements Hygro-Thermal Properties





### Design Requirements Environmental Properties

Fatigue Strength Fatigue Degradation Crack Propagation Characteristics Vibration Degradation Shock / Impact Degradation	
Fatigue Degradation Crack Propagation Characteristics Vibration Degradation Shock / Impact Degradation	
Crack Propagation Characteristics  Vibration Degradation  Shock / Impact Degradation	
Vibration Degradation Shock / Impact Degradation	>
Shock / Impact Degradation	>
Atomic Owygen Attack	` <b>\</b>
CANGE CANGER ARREST	>
UV Degradation	>
Corresion	<b>&gt;</b>
Micrometeoroid Erosion	<b>\</b>

#### Design Requirements Other Considerations

Orbital Sciences Corporation



Availability of Various Forms Safety / Health Concerns Availability for Schedule Handling Concerns Manufacturability Heritage Cost

OSC / Space Systems Division 21700 Atlantic Boulevard Dulles, Virginia 20166 (703) 406-5000

### DESIGN of COMPOSITE SPACE STRUCTURES

SDIO WORKSHOP on Graphite/Polycyanate

MIKE DEAN BALL AEROSPACE (303) 460-3748

## SPACE STRUCTURES WORKSHOP



- ACCEPTANCE CRITERIA for COMPOSITES
- MATERIAL REQUIREMENTS
- SPACECRAFT SYSTEM REQUIREMENTS
- PROPOSED DEM/VAL ACTIVITIES

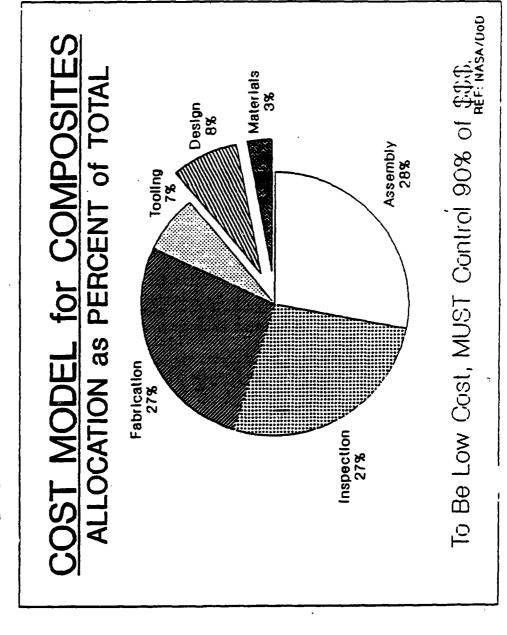


### SPACE STRUCTURES WORKSHOP ACCEPTANCE CRITERIA for COMPOSITES

- INDUSTRY THRUST ---> SmallSats are needed.
- Full Mission Capability
- High Efficiency and Performance
  - Cost Effective Operations
- ESTABLISH SPACE FLIGHT HERITAGE
- MINIMIZE program risk
- DEMONSTRATE operational benefits
- OFFER credible solutions for technical superiority
  - SATISFY political realities

### Low Cost Access to Space

## High part costs due to inefficient processing





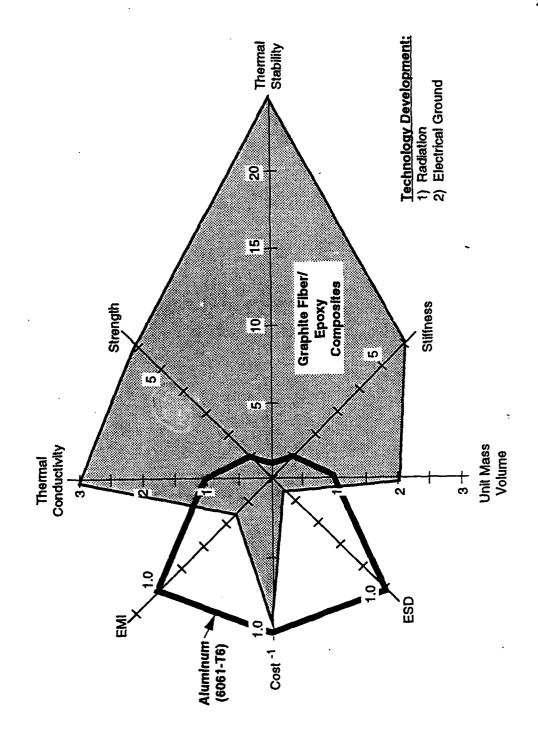


### SPACE STRUCTURES WORKSHOP MATERIAL REQUIREMENTS

- Mechanical properties:
- 1) Strength allowables for minimum weight
- First ply failure
- Residual strength at ultimate failure
- 2) Thermal and moisture expansion, out-gassing (NASA) 3) Impact from debris and micrometeoroid 4) Enhance compatibility with high-modulus fibers
- Enhance compatibility with high-modulus fibers
- 1) Thermal control 2) EMI/ESD protection 3) Resistance 1 Integrated system design:
- Resistance to space environments (service life)
- Versatile fabrication and assembly:
- 2) Adjust to configuration evolution (long design cycle) 3) Compatible adhesive and and adhesive adhesive and adhesive adhesive and adhesive and adhesive and adhesive and adhesive and adhesive adhesive and adhesive adhesive and adhesive and adhesive and adhesive adhesive and adhesive adhesive adhesive and adhesive adhesive
  - Compatible adhesive grades of resin system
- Performance: COMPOSITES > ALUMINUM



### Design Versatility With Multi-Functional Composite Structures





### SPACE STRUCTURES WORKSHOP SPACECRAFT SYSTEM PERFORMANCE

- SATISFY RELIABILITY ISSUES
- MINIMIZE risk for Program Manager
  - \* Cost
- · Schedule
- ESTABLISH flight heritage for advanced technology
  - New capabilities
- Maintain competitiveness
- REDUCE RISK with GROUND QUALIFICATION TESTING
- Material characterizations
- Component and subassembly functional tests
- · System-level vibration and thermal vacuum tests
- VERIFY with SPACE FLIGHT EXPERIMENTS



### SPACE STRUCTURES WORKSHOP PROPOSED DEM/VAL ACTIVITIES

- DEVELOP COMPATIBILITY with EMERGING TECHNOLOGIES
- · Smart Structures
- Autonomous Control Concepts
- Design Optimization for MINIMUM Weight
- IMPLEMENT NON-STRUCTURAL DESIGN STANDARDS for COMPOSITES
- Understand EMI/ESD behavior
- Define empirical relationships for design
- Develop analytical codes for multi-component, anisotropic material systems
- VERIFY with FLIGHT EXPERIMENTS

### WORKSHOP ON GRAPHITE-REINFORCED POLYCYANATE COMPOSITES

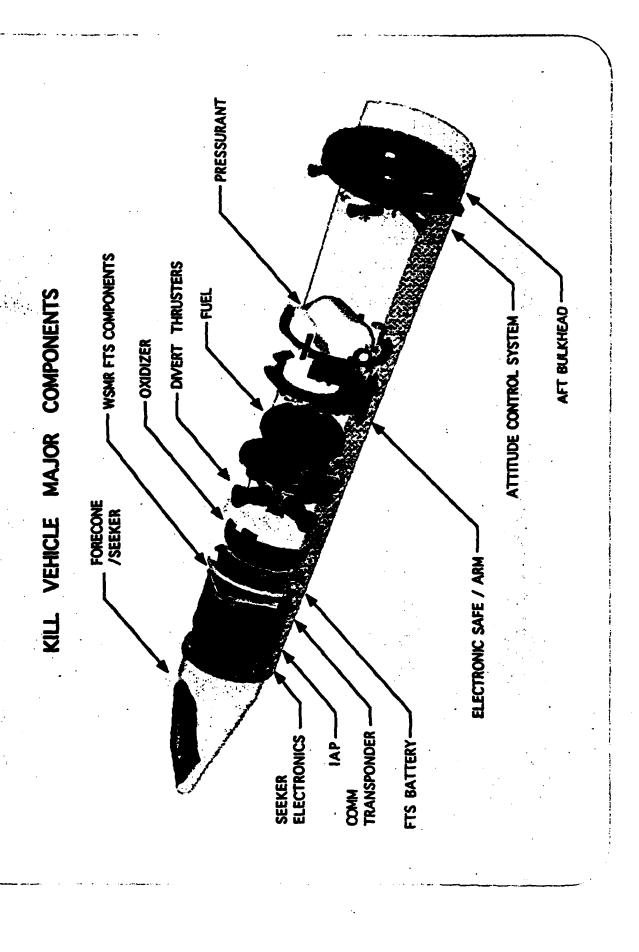
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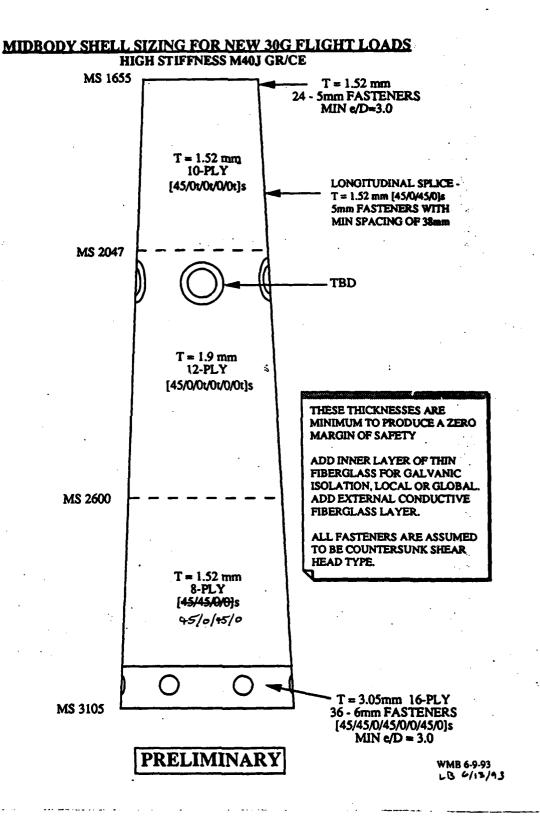
Gordon Ritchie O/74-12, B/551

LOCKHEED MISSILES AND SPACE COMPANY
P.O. BOX 3504
SUNNYVALE, CA 94089
(408) 743-1588

#### AGENDA

- THAAD APPLICATIONS
- SPACECRAFT APPLICATIONS
- GENERAL CONCERNS
- SPECIFIC APPLICATION CONCERNS
- MATERIALS OF INTEREST
- SUMMARY





### THAAD APPLICATIONS

(Theater High Altitude Area Defense)

Quantity

Shelf Life

**Hundreds to Thousands** 

20 Years

5+ Minutes

Up to 550°F

Composite Laminate Design

Operating Temperatures

Operating Life

Moderate to High Strength Moderate to High Stiffness

## SPACECRAFT APPLICATIONS

Quantity

One to Many

• Shelf Life

Varies

Operating Life

5+ Years

Operating Temperatures

-250° to +250°F

Composite Laminate Design

High Stiffness, Moderate Strength

Contamination Requirements
 Outgassing

Yes

Dimensional Stability Requirements

Yes

-CTE

-CME

- Microcracking

### GENERAL CONCERNS

- Lack of Material Property Design Database
- Lack of Joint Property Design Database
  - Bonded
    - Bolted
- Limited USA Flight Experience
- Foreign Supplier (Fibers)
- Amoco Exit from Prepreg Business
- "Tack" Property Lower than Epoxy and for Less Time
- Resin Cost about Four Times Higher than Epoxy

## SPECIFIC APPLICATION CONCERNS

• THAAD

-Effects of Post-Cure on Properties

Spacecraft

-Dimensional Stability

CTE

CME Microcracking - Contamination Due to Outgassing

Lack of Thin (<2-Mil) Prepreg with Ultra-High Modulus Fibers

### **MATERIALS OF INTEREST**

• MATRICES

Hexcel F475

YLA RS-3Amoco 1939-3Fiberite 954-2AFiberite 954-3

FIBERS

T300 M40J M60J P75 P120

### SUMMARY

CYANATE ESTER MATERIAL PROPERTIES DATA ARE NEEDED

## Graphite-Reinforced Polycyanate Composites

### DESIGN PERSPECTIVE

Government/Industry Workshop on Graphite-Reinforced Polycyanate Composites

at

Institute for Defense Analyses Alexandria, Virginia

June 16, 1993

Bobby Hanson (303)971-9394

## Why Gr/Pc for Space Structures?

### LOW PROPERTIES

Outgassing

Moisture Absorption/Strain

Microcracking (Thermal Cycling, Radiation)

Dielectric Constant and Loss Tangent

### HIGH PROPERTIES

**Toughness** 

Glass-Transition Temperature

## REAL REASONS TO USE GR/PC FOR SPACE STRUCTURES

"Good Old Boy" Processing

Get the Above Nice Things at Negligible Cost Impact

### MARTIN MARIETTA

### MARTIN MARIETTA

## **Environments and Material Design Drivers**

### INTEGRATION/TEST/HANDLING

- -Rarely a Material Design Driver
- -Material Toughness is Needed Because People Are Involved

#### LAUNCH

- -Usually Governs Structural/Material Design
- -Stiffness, Strength, Low Cycle/High Amplitude Fatigue

#### **OPERATION**

- -Governs Some Aspects of Structural/Material Design
- -Requirements Mission Specific

## Precision Truss Structures REQUIREMENTS

-Launch/Operational Stiffness

-Launch Strength

-Dimensional Stability

High Modulus

**PAN Fibers** 

Near-Zero CTE

Low Hygroscopic Strain (β \* m)

Low Microcracking (Thermal Cycling, Radiation)

Polycyanates

Creep Resistance

-Low Outgassing

## Non-Precision Box Structure REQUIREMENTS

-Launch Stiffness -Launch Strength

### Atmospheric Entry Forebody REQUIREMENTS

-Launch Strength

-Entry Strength (Temperature Up To 500 F) -Entry Stiffness (Temperature Up To 500 F)

### Thermal Management Components REQUIREMENTS

- -Launch Strength -High Thermal Conductivity

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# Issues. or How Do You Get Gr/Pc Into a Svstem?

### QUALIFICATION TESTING

- -Approach Varies Greatly
- -Complete Database Greatly Increases Chance of Being Used

### MATERIAL MATURITY

- -Heritage Invaluable for Developing User Confidence
- -Stability in Product Forms and Availability
- -Product Consistency
- -Stability in the Marketplace
- -Have All the Issues Surfaced?

### MARTIN MARIETTA

### Conclusions

Polycyanate Resins Have Numerous Favorable Attributes

Performance Advantages Are In General Evolutionary, Not Revolutionary

Payoffs Are More Qualitative than Quantitative, and Take Forms Such as Increased Robustness and Reliability Polycyanates Must Remain Cost Competitive with Other Resins to Survive

## IDA Workshop on Graphite-Reinforced Polycyanate Composites

# APPLICATIONS OF GRAPHITE-REINFORCED COMPOSITES

## THE LORAL PERSPECTIVE

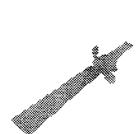
### JOHN COONEY

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## Our Major Programs



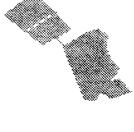
INTELSAT



GOES



**N-STAR** 



Globalstar

SUPERBIRD

Space Station

Materials Engineering

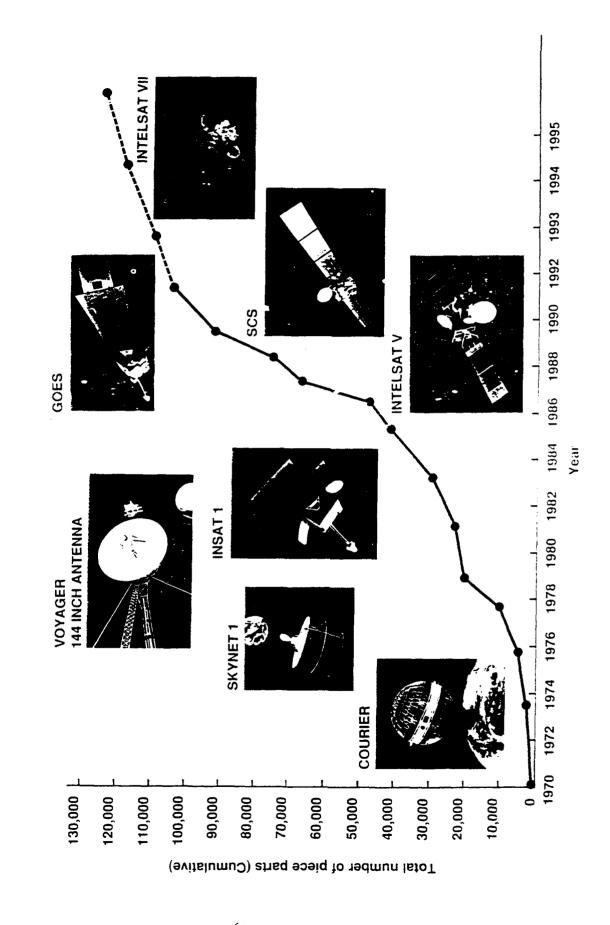
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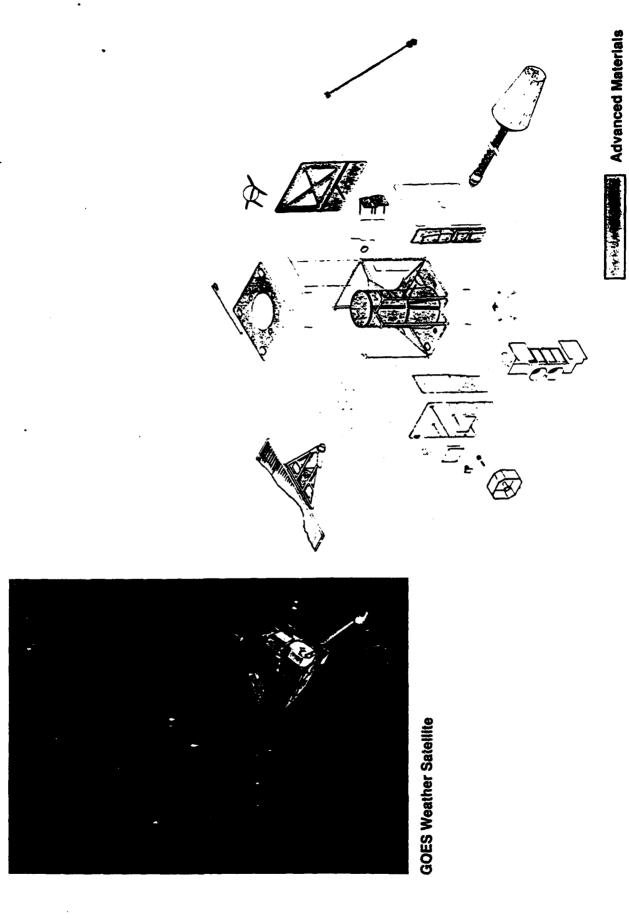
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# THE LEADER IN ADVANCED MATERIALS APPLICATIONS IN SPACE



# ADVANCED COMPOSITE MATERIALS APPLICATIONS - GOES......

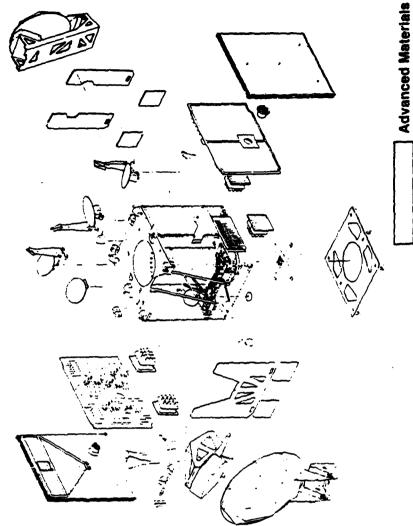


### SPACE BYSTEMS/LORAL

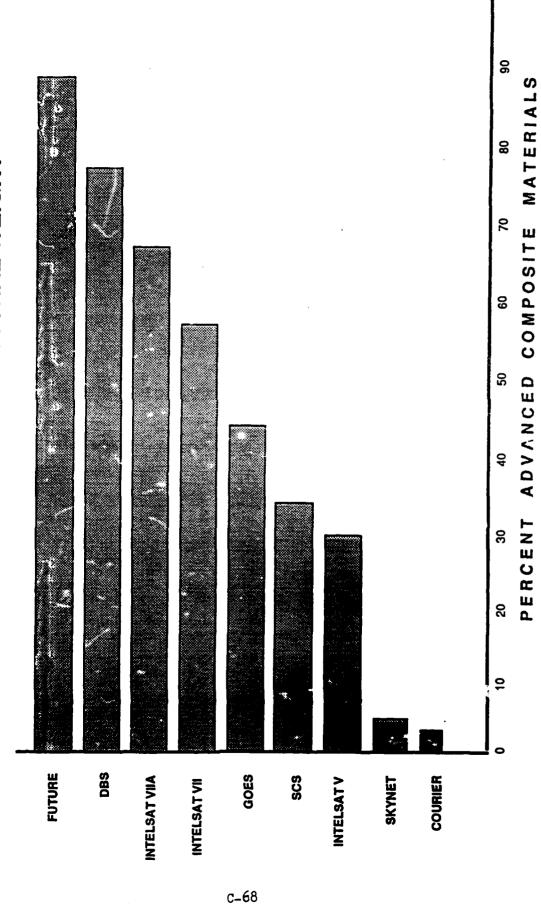
# ADVANCED COMPOSITE MATERIALS APPLICATIONS - INTELSAT VII......



INTELSAT VII Communications Satellite



### ADVANCED COMPOSITE MATERIALS UTILIZATION VS SPACECRAFT STRUCTURAL WEIGHT



## Implementation at SS/L

- IRAD testing in 1988
- Flight hardware materials specifications 1991
- Flight hardware fabrication initiated 1991

c-69

First flight INTELSAT late 1993

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# IDA Workshop on Graphite-Reinforced Polycyanate Composites

### **CURRENT SYSTEM**

#### ICI 934 EDOXY

#### Issues

#### Desires

- Heritage
- Low Outgassing
- Non-toughened
  - High Moisture Absorption
- Microcracking

Ease of Processing

High Tg

- "Toughened"
- Reduced Moisure Absorption
- Higher Tg
- Little/no Process Changes
- Temperature Lower Cure

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### NEW SYSTEM(S)

Polycyanate Resin System(s):

- Can be Toughened
- Have Reduced Microcracking
- Have Reduced Moisture Absorption

C-71

- Have Higher Tg
- Process like ICI 934 Epoxy (350° Cure)
- Exhibit Low Outgassing

## Comparison of Typical Resin Properties

	I Vpical" Polycyanate	ICI 934 EDOXY
Tg	~250°C*	200°C
Service Temperature	350°F	350°F
Specific Gravity	1.19*	1.30
Maximum Moisture Content	1.2%*	%0.9
Tensile Strength	10 ksi*	4.0 ksi
Tensile Modulus	470 ksi	600 ksi
Processing Characteristics	Similar to 934	
Outgassing	Passes E-595	Passes E-595

C-72

\* Key Advantages

# Materials Qualification Test Plan

#### **Materials Tested**

Supplier

Composite System

• AMOCO

K-1100/1939-3

Tape

Fabric

Tape Tape

Fiberite

XN50A/9:34-2A XN50A/954-A XN70A/954-2A MJ60/954-2A MJ60/954-2A

Tape Fabric

¥7-+06/000

Kevlar 49/RS-3

XN50A/RS-3

Fabric Tape

> XN50A/RS-3 Quartz/RS-3

Tape Fabric Fabric

YLA

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# Materials Qualification Test Plan

### Mechanical Properties

SI	
9	

#### Method

0° Tensile Strength, Modulus, Poisson's Ratio ASTM D 3039 (SRM 4-88)

90° Tensile Strength, Modulus

**ASTM D 3039 (SRM 4-88)** 

0° Compression Strength, Modulus

**ASTM D 3410 (SRM 1-88)** 

In-plane (± 45°) Shear Strength, Modulus

ASTM D 3518 (SRM 7-88)

Flexural Strength, Modulus

ASTM D 790

Short Beam Shear

ASTM D 2344 (SRM 8-88)

**Environmental Testing** 

+100°C -100°C

All of the above except for flexural and short beam shear tests

Laminate Properties

Fiber volume

ASTM D 3171 (Proc. C) **ASTM D 2734** 

Void Content

**ASTM D 792** 

Laminate Density

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# Materials Qualification Test Plan

### Physical Properties

Method

Test

IR scan of uncured resin

Uncured resin content

Volatile content

Resin flow

SPI-prepreg-2, Test C

**ASTM D 3530** 

**AMS 3903** 

SPI-prepreg-3

**ASTM E-595** 

Gel time

Outgassing

CTE 0° and 90°

CME 0° and 90°

Uncured and cured ply thickness

-180°C to +135°C, then tested at room temperature Thermal cycling - Minimum 1 lot of material, cycled 10 times from for 0° Compression Strength and Modulus.

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# IDA Workshop on Graphite-Reinforced Polycyanate Composites

# Room Temperature Data Summary

	M60J/954-2 Tape	M60J/954-2 Fabric	XN50-954-2A Fabric	XN50/RS-3 Fabric	XN70A/954-2 Tape	K1100/1939-3 Tape	Kevlar 49/RS-3 Fabric	
0° Tensile Strength (ksi)	272.0	110.4	113.2	122.7	‡ •	140.5	6.69	
0° Tensile Modulus (msi)	50.7	25.5	25.9	27.2	. !	74.6	4.6	
90° Tensile Strength (ksi)	3.5	77.1	85.6	129.8	i		1	
90° Tensile Modulus (msi)	6.0	27.1	25.7	26.4	ŧ	į	ł	
Compressive Strength (ksi)	9.68	50.1	29.4	29.4	51.1	39.5	20.9	
Compressive Modulus (msi)	43.6	20.2	12.9	16.1	56.2	80.6	5.3	
In-Plane Shear Strength (ksi)	7.5	10.3	15.6	14.9	8.1	4.8	7.9	
In-Plane Shear Modulus (msi)	0.62	0.63	0.65	0.68	0.62	0.63	0.22	
Flexural Strength (ksi)	125.5	86.2	76.2	80.0	100.3	75.2	47.5	
Flexural Modulus (msi)	41.6	20.9	15.7	15.7	49.6	61.1	4.6	
Short Beam Shear Strength (ksi)	9.6	7.3	7.8	8.7	9.5	3.5	3.7	
Poisson Ratio	0.29	0.029	0.009	0.037	I	0.43	0.050	
Outgassing TML%	0.17	i	0.11	0.095	0.167	0.172	1.187	
VCM%	0.004	i	0.001	0.004	0.000	0.002	0.055	
Fiber Volume (%)	61.8	59.5	60.3	0.09	59.9	56.1	60.3	

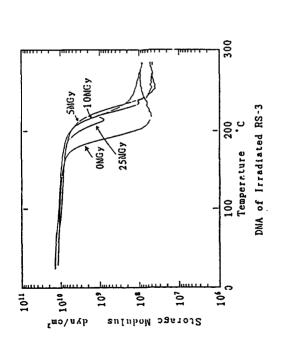
All data except for SBS normalized to 60% fiber volume

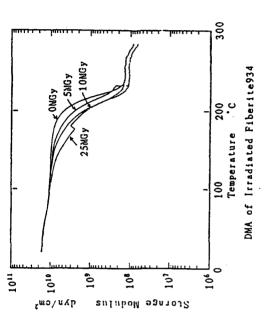
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### Simulated Space Environmenta **Fest Data**





#### **Electron Beam Irradation**

Source: Nippon Petrochemical Co. LTD

(10 MGy corresponds to irradation energy of 3 years at synchronous orbit.)

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## HARDWARE FABRICATED

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16' Foldable Reflector

**Battery Sleeves** 

C-Band Feed Horns

Central Cylinder

Radiator Panels

Solar Array Yoke

Composite Hinge High Power Mux

Graphite Honeycomb core

#### **Application** Material

IRAD XN50/RS-3

-VII, I-VIIA, N-Star...

K1100/1939-3

V-Star...

N-Star...

M60J/954-2

XN50/RS-3

IRAD K1100/1939-3

IRAD, N-Star

M60J/954-2

IRAD... K1100/1939-3

IRAD... XN50/RS-3

IRAD XN50/RS-3

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## FABRICATION ISSUES

- Simple Contour Parts
- Problems less than 934
- Multiple Contour Parts
- Problems with lack of tack
- May require additional debulk operations
- No "show stoppers"

## Recommended R&D Directions

- Additional characterization and optimization of effects of moisture and thermal cycling on dimensionally stable structures.
- effects Additional characterization of radiation graphite-reinforced polycyanate composites.
  - Characterize LEO effects on polycyanates.
- Develop a low temperature cure system with Tg >> 300°F.
- Characterize/evaluate polycyanate adhesives for structural bonding applications.
- Additives to polycyanates for enhanced thermal and electrical performance.
- Further characterization of fracture toughness and other design criteria.

#### CREDITS

**AMOCO** 

**Fiberite** 

**Marshall Consulting** 

Nippon Petrochemicals Company, LTD

YLA

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## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

Bill Harvey / 310-797-4870

#### BRIEFING CONTENTS

- o Basic Material Selection Guidelines
- Rockwell Sensorcraft Baseline Structural Materials 0
- Tell Us All The Good Stuff About Why Polycyanate Is Better 0
- Roadblocks & Detours Encountered on The Road to a Hardware Program Product 0
- o Suggested Solutions to These Show Stoppers

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## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

Bill Harvey / 310-797-4870

## **BASIC MATERIAL SELECTION GUIDELINES**

- and pleaded with them to use graphite to make their spacecraft lighter, stiffer, PROJECT MANAGEMENT brought bags of Gold to the STRUCTURES PEOPLE Some day our Greatgrandchildren will hear stories about the days when and more dimensionally stable than ever before. 0
- o Those days are gone.
- Today, in an effort to meet customer performance requirements at: 0
- the lowest material cost,
- lowest risk,
- lowest cost to design and analyze,
- and lowest cost to fabricate & assemble,

the trend is to avoid the use of advanced materials unless forced into it.



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Bill Harvey / 310-797-4870

#### STRUCTURAL SYSTEM IS PREDOMINATELY DEFINED THE ROCKWELL SENSORCRAFT BASELINE BY THE MATERIAL: ALUMINUM.

#### The Aluminum Structural Load Paths satisfy all: 0

- manufacturing cost & schedule,

C = 84

- structural strength and stiffness,
- weight,
- heat transfer,
- litter response,
- outgassing and moisture absorption,
- UV degradation, Radiation, and EMI

requirements for all the subsystems defined by it.



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## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

Bill Harvey / 310-797-4870

### ADVANCED MATERIALS TO MEET SENSORCRAFT REQUIREMENTS. WE DO HAVE A FEW UNIQUE APPLICATIONS WHICH REQUIRE

The optimum material for both facesheets and stiffeners of a sandwich structural subsystem has been determined to be 6090/SiC/25P PMMC. 0

"It's just like Aluminum."

- Two other types of subsystems require a moderately high modulus composite such as provided by AMOCO's P75/1939-3 tape. 0
- The first subsystem type are struts defined by an orthotropic ply layup which provide the optimum stiffness to weight ratio required to meet first mode frequency prerequisites. 0

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## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

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#### THE OTHER IS A LIGHT WEIGHT ASSEMBLY OF SANDWICH PANELS

- Some of the panels are designed to react both twisting and bending. 1.50 in. deep 1.0 Lb/Ft³ Hexcel honeycomb core is sandwiched by [0/±60], P75/1939 facesheets. 0
- Others are designed to react bending and they also are defined by 1.0 Lb/Ft3 core and P75/1939 facesheets. 0
- Closeouts and local doublers for these panels are defined by T300/1939 lamina. 0
- Support Beams are made up of an orthotropic ply layup of P75/1939 which provides the bending stiffness needed to meet the first mode frequency requirements of this subsystem.

0



Held At The Institute for Defense Analysis - Alexandria, VA. on June 16, 1993



## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

Bill Harvey / 310-797-4870

### IGHT WEIGHT PANEL ASSEMBLY IS DESIGNED TO MEET JITTER, ATTITUDE CONTROL, AND CONTAMINATION REQUIREMENTS

- Attitude control requirements dictate a light weight Panel Assembly design. 0
- required to 'tune' the subsystem frequencies, but at the low density demanded by P75 fibers provide an inexpensive means for obtaining moderately high stiffness, this design. 0
- P75/1939 is available in .0015 in. thick lamina, which enable the design of thin ply facesheets with small constitutive equation coupling terms. 0
- AMOCO's 1939 modified epoxy matrix exhibits comparatively low microcracking, outgassing, and water absorption. 0
- But most important, it is a matrix that provides a good interfacial bond between the fibers and matrix. Previous experience with other Pitch based fibers and epoxy matrix systems has resulted in careful application and mixing of the P75/Epoxy composite due to Bimodular material properties.

0



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## TELL US ALL THE GOOD STUFF

## **ABOUT WHY POLYCYANATE IS BETTER**



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#### On day two of the Feb. 13, 1992 Phillips Lab Adaptive Structures Review held at TRW, TRW's George Dvorsky reported on some promising Polycyanate material characteristics.

TABLE 1. Polycyanate vs Epoxy Matrix Material Properties Critical to Sensorcraft and Lightweight Stable Spacecraft Subsystems Such as Large Graphite Reflectors.

MATERIAL PROPERTY	POLYCYANATE	EPOXY	XX
	(Ref. TRW AMASS VG)	Generic Epoxy (Ref. TRW AMASS VG)	P75/1939-3 Rockwell B.E. Baseline (Ref. AMOCO Data Sheets)
Moisture Absorption	> 1.5%	%4	2%
Coefficient of Moisture Expansion (ppm/%)	149	261	NA
Outgassing TML: VCM:	0.20% 0.01%	1.00% 0.07%	0.18% 0.00%
Thermal Cycle Induced Microcracking (Microcrack Density cm <sup>-1</sup> @ 50 Cycles)	2.50	21.5	1.0



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TABLE 2. Polycyanate vs Epoxy Matrix Elastic and Strength Material Properties.

MATERIAL PROPERTY	POLYCYANATE	EPOXY	XX
	(Her. 1HW AMASS VG)	Fiberite 934 (Ref TRW AMASS VG)	AMOCO ERL-1939-3
Tensile Strength (KSI)	12	4	NA
Tensile Modulus (KSI)	430	009	200
Strain to Failure (%)	4.9	7.	NA
Tg (°F) (post cured)	490	385	464
Density (g/cm³)	1.19	1.30	1.23

#### AMOCO modified epoxy. Other than the strain to failure, what other advantages There doesn't appear to be much difference between the Polycyanate and the and or disadvantages are there? 0

- Cost?
- Impact Damage Tolerance?
- Damping?
- Nuclear Dose and Atomic Oxygen Environmental Data to Predict Long Term Structural/Thermal/Physical Stability?
  - Long Term (>5 years) Material Degradation in high U.V. Environment?
- Poor pan based fiber to matrix interfacial bond resulting in Bimodular Material Properties?
- Adhesive or Mechanical Joining Issues?
- ETC.

### Anybody have MATERIAL QUALIFICATION TEST results for P75/Pc & P100/Pc? 0

UNCLASSIFIED

Space Systems Division





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### ROADBLOCKS & DETOURS ENCOUNTERED Bill Harvey / 310-797-4870

#### NO

# THE ROAD TO A HARDWARE PROGRAM PRODUCT



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## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

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# ISSUES WITH APPLICATION OF NEW MATERIAL AND HARDWARE

product to meet his needs within the time and budget constraints imposed on the STRUCTURES PEOPLE really do want to provide the customer with the best product development. 0

But there is a good chance that he can't because of the rules that he must work 0

One preliminary design effort after another over the past few years has been hampered by the following scenarios. 0

These scenarios have occurred in virtually all areas of our industry where a new advantage (examples: new lightweight Air Force/Navy Jet Trainer, several recent product requires high performance materials to gain the required competitive spacecraft, and a submersible vehicle). 0

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## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

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### PROGRAM ABC SHOW STOPPER #1

- is degrading heat radiation surfaces, and the manufacturing costs are only a little litter distortions within the error budget, eliminate the outgassing problem which PEOPLE determines that T300/Magic Matrix will save hundreds of pounds, bring Finite Element Aided Optimization Analysis performed by the STRUCTURES more than the stuff we've used before. 0
- After the cheering dies down, PROJECT MANAGEMENT stands up and says 'Bill, budget looks like, I just don't have the schedule and money to put together a you know that they keep cutting back on the funding and you know what my MATERIAL QUALIFICATION PROGRAM for this stuff." 0
- End of discussion, end of the customers performance advantage, and end to the chance to incorporate the technology in future efforts. 0



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## ROCKWELL SENSORCRAFT STRUCTURAL SYSTEM DESIGNER PERSPECTIVE

Bill Harvey / 310-797-4870

# SUGGESTED SOLUTION TO 'MATERIAL QUALIFICATION' ISSUE

The CUSTOMER TECHNOLOGY SUPPORT AGENCY is Pretty Smart. Before the CUSTOMER even thinks about issuing the Program ABC RFP, the AGENCY knows what material characteristics and materials are required to produce the product.

0

- STRUCTURES PEOPLE keep their eyes open too, and are usually aware of unique material characteristics that will give their designs a competitive edge. 0
- STRUCTURES PEOPLE by inviting them to workshops where he discusses the merits of The CUSTOMER TECHNOLOGY SUPPORT AGENCY plants seeds in the minds of the some of the materials he feels will give Hardware Program ABC a competitive edge. 0
- MATERIAL per a unique Manufacturing Process Specification that meets programmatic It is suggested that the CUSTOMER TECHNOLOGY SUPPORT AGENCY QUALIFY THE 0
- The CUSTOMER through the AGENCY will provide these test results and material dependent design characteristics to Hardware Program ABC STRUCTURES PEOPLE upon request 0
- When THE CONTRACTOR wins Hardware Program ABC, because of the superior design and lowest cost, the CUSTOMER will then provide the Manufacturing Process Specification to THE CONTRACTOR'S MANUTACTURING PEOPLE, so it can be built. 0

UNCLASSIFIED

## THE APPLICATION OF GRAPHITE POLYCYANATE COMPOSITES TO TO FLIGHT HARDWARE

PRESENTATION TO THE WORKSHOP ON GRAPHITE REINFORCED COMPOSITES JUNE 16, 1993

PAM BURNS (310) 364-8462



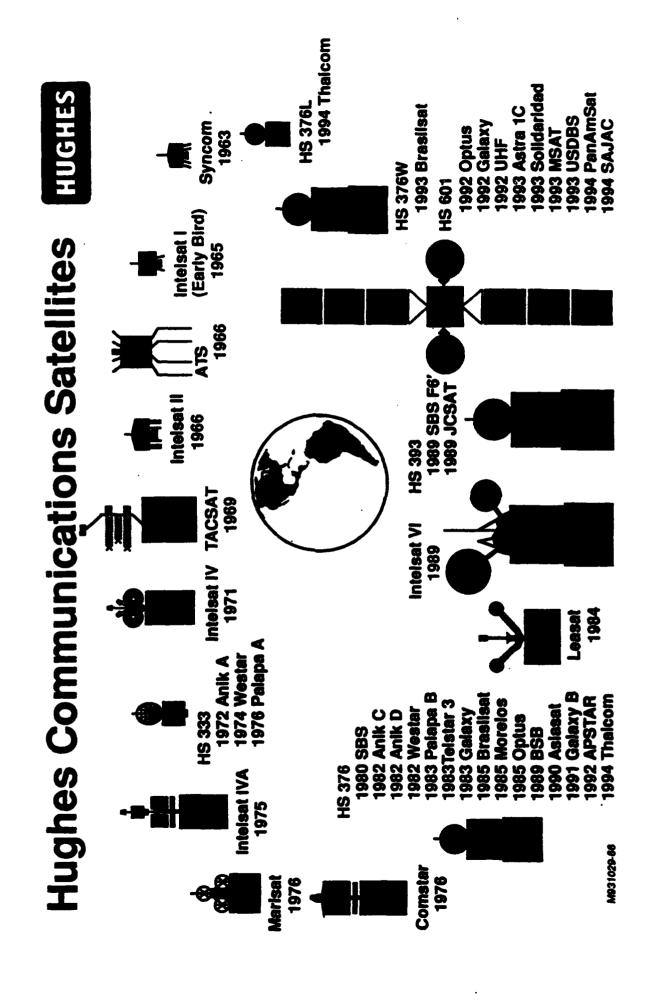
### GMHE Organization



Delco Electronics

HUGHES

G901537-7





### GRAPHITE REINFORCED POLYCYANATECOMPOSITES

## SPACECRAFT DESIGN AND MATERIAL REQUIREMENTS

Design Requirements	Material Requirements
Dimensional Stability	Low Thermal Expansion High Thermal Conductivity Low Hygroscopy Minimal Microcracking
High Structural Rigidity (Specific Stiffness)	High Modulus Low Density Dissimilar Material Compat.
Non-Contaminating	No Outgassing
Rapid Tract and Point Capability	Good Vibrational Damping Characteristics
Deployable	Lightweight Unique Configurations
Survivable in Laser/Nuclear Environments	High Strength No Induced Outgassing High Temperature Integrity Controllable Reflectivity Temp. Dissipation Control
Minimal Space Charging	Good Electrical Conductivity
Low Cost	Manufacture/Producability Versatile Fabrication Efficient Joining & Faster Repairable

#### HUGHES

### GRAPHITE REINFORCED POLYCYANATE COMPOSITES

WHY THE INTEREST IN GRAPHITE POLYCYANATE COMPOSITES?

**CLEANER SPACECRAFT** 

Important for vehicles with optical devices cryogenic coolers, critical thermal finishes and extended orbit time.

REDUCED GEOMETRIC DISTORTION

Improves pointing accuracy of critical structures

REDUCED WEIGHT

Lower resin density. Replacement for metallic designs

**INCREASED TOUGHNESS** 

Increase structural robustness

IMPROVED RADIATION RESISTANCE

**Extend structural life** 

HARDWARE BY TAKING ADVANTAGE OF SIMILAR HANDLING AND THESE BENEFITS CAN EASILY BE TRANSITIONED TO FLIGHT PROCESSING CHARACTERISTICS TO EPOXY RESINS.

#### HUGHES

### GRAPHITE REINFORCED POLYCYANATE COMPOSITES

SPACECRAFT APPLICATIONS:

STABLE STRUCTURES

TRUSS STRUCTURES

**SOLAR PANELS** 

**ANTENNA REFLECTORS & ARRAYS** 

**EQUIPMENT SHELVES & SUPPORTS** 

LIGHTWEIGHT NONCONTAMINATING STRUCTURES

**OPTICAL BENCH** 

**SOLAR PANELS** 

**METALLIC REPLACEMENT (ALUMINUM AND BERYLLIUM)** 

STRUTS

**ELECTRONICS PACKAGING** 

MISSILE STRUCTURES

RADAR STRUCTURES

**OPTICAL STRUCTURES** 

### GRAPHITE REINFORCED POLYCYANATE COMPOSITES

HUGHES

HUGHES OBJECTIVE IS TO RAPIDLY IMPLEMENT THE GRAPHITE PROPERTIES POLYCYANATES BY TAKING ADVANTAGE OF "EPOXY" LIKE PROPERTIES

TYPICAL APPLICATIONS OF HIGH MODULUS GRAPHITE REINFORCED **POLYCYANATES** 

**BUILDING P100/1939 HARDWARE TO FLY IN EARLY 1994** 

P75/934 ADAPTOR AND STRUTS BUILT FOR AIR FORCE

T650/1939 EQUIPMENT SHELVES

**SYSTEMS TESTING K1100/1999** 

**QUALIFICATION TESTING XN 70/RS3** 

P120/954 GBI STRUCTURE

MATERIAL SUPPLIERS AND FABRICATORS FOR "CONCURRENT" HUGHES APPROACH TO DESIGN DEVELOPMENT IS TO TEAM WITH ENGINEERING.

#### GRAPHITE REINFORCED POLYCYANATE COMPOSITES



**NUMBER OF COUPONS TESTED TO PRODUCE B ALLOWABLES (TYP. 16) HUGHES MINIMUM REQUIRED DATA BASE FOR DESIGN ALLOWABLES: DESIGN ALLOWABLES TYPICALLY DEFINED AS STRESS - 2(STD) MINIMUM 2 DISTINCT LOTS OF PREPREG TESTED** 

**TESTING PERFORMED IN THREE STAGES:** 

QUALIFICATION TESTING (RESULTING IN A PROCUREMENT SPEC)

- **GENERATE DESIGN DATA BASE**
- PERFORMED TO VERIFY WETABILITY AND TRANSLATION OF PROPERTIES

SYSTEMS LEVEL TESTING (SPECIFIC FOR INTENDED OR GENERIC **APPLICATION)** 

PERFORMED ON SUBELEMENT OR FULL SCALE PROTOTYPE FIRST FLIGHT ARTICLE QUAL TEST (CRITICAL APPLICATIONS)

#### HUGHES

### GRAPHITE REINFORCED POLYCYANATE COMPOSITES

### MATERIAL QUALIFICATION PROGRAM

### **MECHANICAL PROPERTIES**

- TENSILE STRENGTH, MODULUS AND POISSONS RATIO (ASTM D3039) **LONGITUDINAL AND TRANSVERSE**
- **COMPRESSIVE STRENGTH AND MODULUS (ASTM D3410)**
- **INPLANE SHEAR STRENGTH AND MODULUS (ASTM D3518) LONGITUDINAL AND TRANSVERSE**
- **INTERLAMINAR SHEAR (ASTM D2344)**
- FLEXURAL STRENGTH AND MODULUS (ASTM D790)

#### **ADHESION PROPERTIES**

- · LAP SHEAR
- · FLATWISE TENSION

#### GRAPHITE REINFORCED POLYCYANATE COMPOSITES

#### PHYSICAL PROPERTIES

- OUTGASSING (ASTM E595)
- CTE (LASER INTERFEROMETRY)

LONGITUDINAL, TRANSVERSE AND QUASI-ISOTROPIC

- MOISTURE ABSORPTIVITY/MICROSTRAIN (70% TO 0% RH)
  - ELECTRICAL CONDUCTIVITY
- THERMAL CONDUCTIVITY
- SPECIFIC GRAVITY (ASTM D792)
- RESIN CONTENT (ASTM D3171)
  - GEL TIME (ASTM D3532)
- VOLATILE CONTENT (ASTM D3520)
- SHELFLIFE
- GLASS TRANSITION TEMPERATURE
- FIR
- FIBER AND NEAT RESIN CERTIFICATION (SUPPLIED BY VENDOR)

• FIBER VOLUME (ASTM D3171)

VOID CONTENT

• RESIN FLOW (ASTM D3531)

· TACK

### HUGHES

# GRAPHITE REINFORCED POLYCYANATE COMPOSITES

### SYSTEMS LEVEL TESTING

### **APPLICATION TESTING**

- BEARING TESTS
- HONEYCOMB SANDWICH TESTS

FLEXURAL STRENGTH AND MODULUS

- THERMAL CYCLING (ENVIRONMENT DICTATED BY APPLICATION TYP. 24 CYCLES -40 TO 80C AS DEFINED BY MIL STD 1540B FOR INTERNAL SPACE STRUCTURES)
- DYNAMIC LOADING

### **UNIT LEVEL TESTING:**

ASSEMBLED UNIT TESTED TO FAILURE



# GRAPHITE REINFORCED POLYCYANATE COMPOSITES

**ADDITIONAL DESIGN INFORMATION NEEDED:** 

ATTACHMENT CONCEPTS

MACHINING

THERMAL EXPANSION MISMATCH

**QUALITY CONTROL** 

**NONDESTRUCTIVE EVALUATION (NDE)** 

SURVIVABILITY

RADIATION

ELECTROSTATIC DISCHARGE POWER GROUND (ELECTRONICS)

REPAIR



# GRAPHITE REINFORCED POLYCYANATE COMPOSITES

## **CURRENT TECHNOLOGY GAPS:**

• LONG TERM CREEP - LONG TERM STABILITY

**MONATOMIC OXYGEN EROSION - LEO APPLICATIONS** 

**LOWER CURE TEMPERATURE CAPABILITIES:** 

TO REDUCE INTERNAL STRAINS

TO MINIMIZE MANUFACTURING DISTORTIONS

**CRYOGENIC PROPERTIES** 

**TECHNICAL SAFETY REQUIREMENTS** 

**ENVIRONMENTAL EFFECTS** 

ON MANUFACTURING

ON LONG TERM STABILITY

### HUGHES

# GRAPHITE REINFORCED POLYCYANATE COMPOSITES

# MAJOR ROADBLOCKS TO RAPID INSERTION OF TECHNOLOGY

# COST RELIABLE AND PROVEN DATA BASE TECHNOLOGY TRANSFER

### APPENDIX D MATERIAL SUPPLIER PERSPECTIVES



# DOMESTIC SOURCE



- **PUBLIC LAW 102-396**
- 6 OCT 92
- -- MINIMUM 75% COAL AND PETROLEUM PITCH CARBON FIBER PROCURED FROM DOMESTIC **SOURCES BY 1994**
- REGULATION SUPPLEMENT (DFARS) AMENDED PROPOSED DEFENSE FEDERAL ACQUISTION

### Summary: Use of Domestic Fiber

### 11 JUN 93

### Precedent: Restriction on PAN Based Carbon Fiber:

- Public Law passed 1990 requiring the Sec of Def to ensure that 50% of annual DoD requirements for PAN fibers be acquired from domestic sources by FY92.

### - DFAR SUP Clause:

- -- PAN fibers contained in the end product shall be manufactured in the US or Canada using PAN precursor produces in the US or Canada
- -- CO may waive in whole or in part if justified (e.g., if a qualified domestic or Canadian source cannot meet scheduling requirements).
- -- Contractor may request waiver by identifying the circumstances and including a plan to qualify domestic or Canadian sources expeditiously

### Pitch Carbon Fiber:

- Public Law passed 1992 directing Sec of Def to ensure that 75% of pitch carbon fiber acquired for DoD use be procured from domestic sources by 1994.
- DFAR SUP Clause Proposed April 93
  - -- Pitch fibers contained in the deliverable product of contracts are to be comprised of 75% which are procured from and manufactured by domestic sources (US and Canada)
  - -- Requirement may be waived in whole or in part by the CO with the approval of the Head of the Contracting Activity if delivery schedule cannot be achieved due to domestic sources not meeting qualification test. Waiver shall be fully justified and documented.

### Polycyanate Update Week of June 14th

- Dow has been working on cyanate chemistry for almost 15 years beginning in 1978.
- Dow's polycyanate resin has ben produced in the same pilot plant facilities since the mid 1980's.
- plant and the cyanation reaction is conducted in Lonza's pilot plant in Visp, Switzerland. The phenolic precursor is made in our Freeport Tx. pilot
- because of their capabilities and their experience with Lonza was selected by Dow to perform this work cyanogen chloride.

**≱**04

### Polycyanate Update

- Problems occurred in the cyanation process that had A routine production campaign began in Dec 1992. not been encountered in the previous 8-10 yrs.
- Freeport, Tx. May 4 and 5 to discuss progress towards Jan. 1993 to determine the cause of the problem and A major effort was undertaken by Dow and Lonza in implement a solution. A joint review was held in determining the cause of production problems.
- Results of the discussions only eliminated probable causes, they did not identify the actual problem.
- precursor perhaps it somehow ages during storage? The best theory remaining focused on the phenolic

**₹** 

### Polycyanate Update

- An action plan was developed to address the remaining theory.
- · If the plan is successful:
- Lonza will be able to supply Dow in spec polycyanate by mid-July.
- If the plan is not successful:
- A long expensive R&D project would be required by both Dow and Lonza to determine the cause of the problem. This is not economically justifiable by either Dow or
- Therefore, Dow would be forced to exit the polycyanate business.

### Time Zero = May 5 **Action Plan**

1. Schedule and produce phenolic precursor in Freeport, TX

4-6 weeks

Air freight phenolic to Lonza

1 week

3. Lonza preparation

2 weeks

2 weeks

5. Air freight polycyanate to Dow

4. Lonza production of polycyanate

1 week

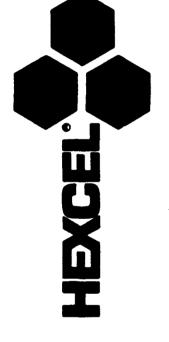
Total time is 10-12 weeks.

We would expect to have polycyanate in our Freeport, TX plant between July 14 and 28.

### Polycyanate Update Summary

- produced almost 6000 lbs of new phenolic precursor and Since the time line was put together on May 5 Dow has air freighted it to Lonza.
- Material was received by Lonza on June 9.
- success of their production efforts in the next 3-4 weeks. Based on current information from Lonza they expect to comply with the May 5th timeline. We will be able to provide the industry with more information as to the
- If there are any questions please contact Chuck Swartz, Dow Chemical, (517) 636-8156.

E HORSE



# CYANATE ESTER PRODUCT LINE

### INSTITUTE FOR DEFENSE ANALYSES **PRESENTATION** 0

## WHY THE INTEREST IN CYANATES

### **ADVANTAGES**

- EXCELLENT ELECTRICAL PROPERTIES
- GOOD MECHANICAL STRENGTHS
- LOW MOISTURE ABSORPTION
- EASILY PROCESSED
- LINEAR CTE
- EASILY MODIFIED

### POTENTIAL DRAWBACKS

- MODEST TO RELATIVELY HIGH COST FOR RESIN
- AVAILABILITY OF BASE MONOMERS
- **EXTENSIVE DATA BASES NOT YET ESTABLISHED**
- "UNKNOWN ISSUES"

# INFORMATION NEEDED FROM DESIGNERS

MECHANICAL PROPERTY REQUIREMENTS

PREFERRED GRAPHITE (PAN OR PITCH) FIBER - ACHIEVE STIFFNESS GOALS - THERMAL MANAGEMENT REQUIREMENTS

TAPE FABRIC PLY REQUIREMENTS

STRUCTURE DESIGN (LAMINATE OR SANDWICH) - COCURABILY ISSUES, NECESSITY FOR FILM ADHESIVE

MICROCRACKING

ENVIRONMENTAL EFFECTS
- THERMAL CYCLING
- RADIATION
- ATOMIC OXYGEN EXPOSURE
- OUTGASSING

PROCESSING LIMITATIONS - CURE TEMPERATURES, NON-AUTOCLAVE,

**COST SENSITIVITIES** 

### HEXCEL CYANATE PRODUCTS

HX1566 - 100% CYANATE RESINON HOLD - PENDING DELETION

DEVELOPMENTAL PRODUCT - PENDING STANDARD PRODUCT STATUS UPGRADE HX1584-4 - 100% CYANATE RESIN

F475 - CYANATE/EPOXY BLEND
STANDARD PRODUCT STATUS (1992)

### HX1566

UNMODIFIED CYANATE-ESTER RESIN SYSTEM
(100% CYANATE). DEVELOPED FOR USE ON JET
PROPULSION LABORATORY PRECISION SEGMENTED
REFLECTOR (PSR) PROGRAM. DATA BASE
GENERATED WITH UHM (HERCULES) FIBER.
POTENTIAL FOR ADDITIONAL SPACE RELATED
APPLICATIONS.

NOTE: CURRENT DRAWBACK RAW MATERIAL AVAILABILITY.

### HX1584-4

UNMODIFIED CYANATE-ESTER RESIN SYSTEM
(100% CYANATE). DEVELOPED FOR RADOME
APPLICATIONS, EXCELLENT ELECTRICAL
PROPERTIES. CURRENT DATA BASE GENERATED
ON QUARTZ AND SPECTRA REINFORCED FABRICS.
CURE CYCLE FLEXIBILITY, 250°F OR 350°F.

SYSTEM ENTERING SCREENING EVALUATION PHASE WITH HIGH MODULUS GRAPHITE FIBER REINFORCEMENTS.

### F475

F475 IS A MODIFIED CYANATE ESTER DESIGNED FOR USE IN SPACE HARDWARE APPLICATIONS. THIS SYSTEM HAS BEEN TOUGHENED AND TRANSLATES HIGH MODULUS GRAPHITE FIBER PROPERTIES EXCEEDINGLY WELL. AN EXTENSIVE DATA BASE IS DEVELOPING VIA QUALIFICATION ACTIVITY FOR SPACE HARDWARE APPLICATIONS. FUTURE PLANS FOR F475 INVOLVE ACTIVE TESTING PROGRAMS ABOARD SHUTTLE EXPERIMENTS.

### CYANATE PRODUCT AVAILABILITY/MATURITY

### **HX1566**

NOT AVAILABLE. OUTLOOK VERY QUESTIONABLE.

### HX1584-4

AVAILABLE. SYSTEM WILL TRANSITION TO A STANDARD PRODUCT STATUS 3/4 QTR. 93. MANUFACTURING PRODUCTION TRIALS NEARING COMPLETION. EVALUATION PHASE WITH HM GRAPHITE REINFORCEMENT BEING INITIATED.

### **F475**

AVAILABLE. STANDARD PRODUCT.
MANUFACTURABILITY PROVEN. DATA BASE
ESTABLISHED AND GROWING. CURRENTLY UNDER
EVALUATION ON THAAD AND EOS.

# FIBER ARCHITECTURES/REINFORCEMENT OPTIONS

CTHICKNESS) CAPABILITY RANGE 70 GMS/M-GMS/M-INCH TO 12 INCH INCH TO 12 INCH ILY USED HAVE BEEN: VMOCO)

BI-DIRECTIONAL FABRIC - ISSUE WEAVABILITY OF FIBER MAY TEND TO LIMIT WEAVING OPTIONS

GENERALLY, IF THE FABRIC CAN BE WOVEN, THE CYANATE PREPREG CAN BE MANUFACTURED

FABRIC WEIGHT RANGES, APPROX. 100 GMS/M<sup>2</sup> TO 400 GMS/M<sup>2</sup>

FIBER EXPERIENCE:

PITCH 120 M40J STANDARD MODULUS

### NEAT RESIN PROPERTIES

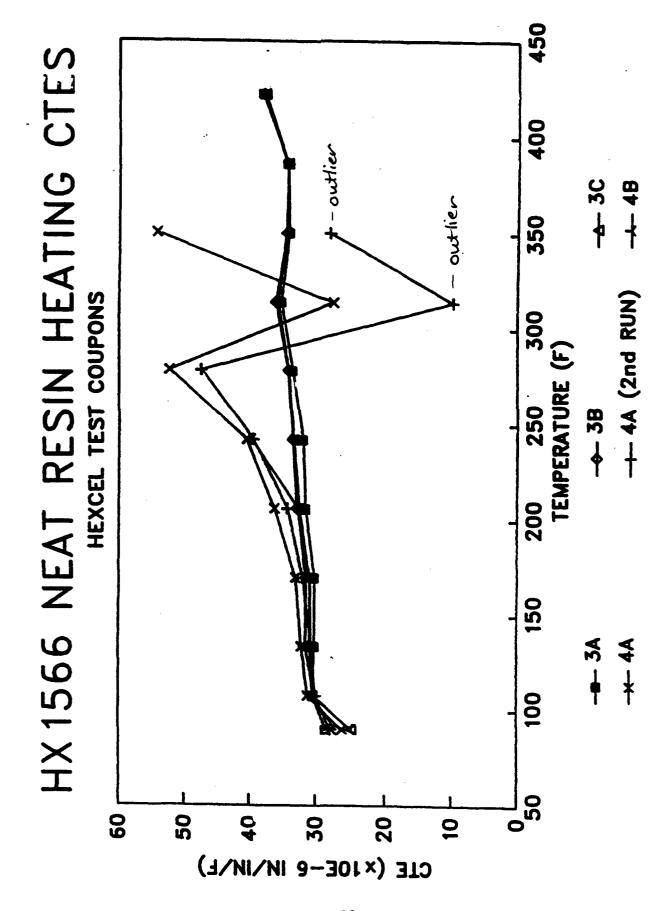
	HX1584-4	F475
TG DRY	396° F	398° F
TG WET	TBD	335° F
MOISTURE ABSORPTION	1.3%	2.0%
FRACTURE TOUGHNESS (K <sub>1c</sub> KSI (IN)	1.2 - 1.6	1.10
DENSITY (GMS/CC)	1.151	1.230
	PREPREG PROPERTIES	
ТАСК	НІСН	Мер-Нісн
OUT-TIME	10 DAYS	3-4 WEEKS
FLOW	НІСН	Moderate
GEL 250° F 300° F 350° F	19:30 MIN 6:45 MIN 1:45 MIN	23:00 MIN 11:30 MIN 5:00 MIN

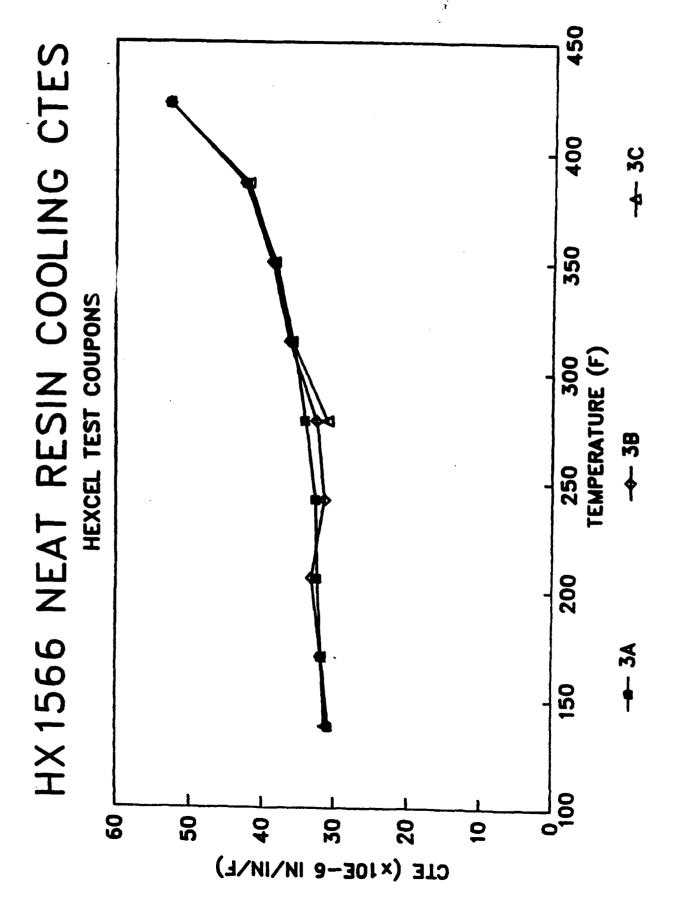
### OUTGASSING DATA

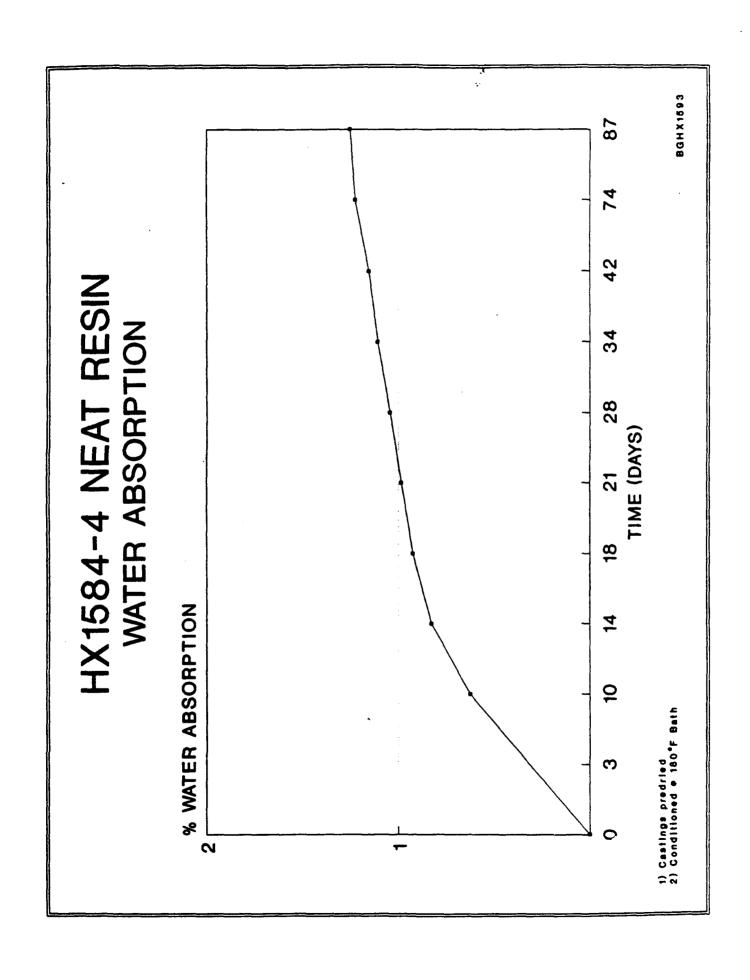
### **NEAT RESIN**

	F475	HX1584-4
TOTAL MASS LOSS (TML):	0.62	0.41
COLLECTED VOLATILE CONDENSABLE MATR'L (CVCM):	0.00	0.00
WATER VAPOR RECOVERED (WVR):	0.31	0.31
APPEARANCE AFTER TEST:	NO CHANGE	NO CHANGE

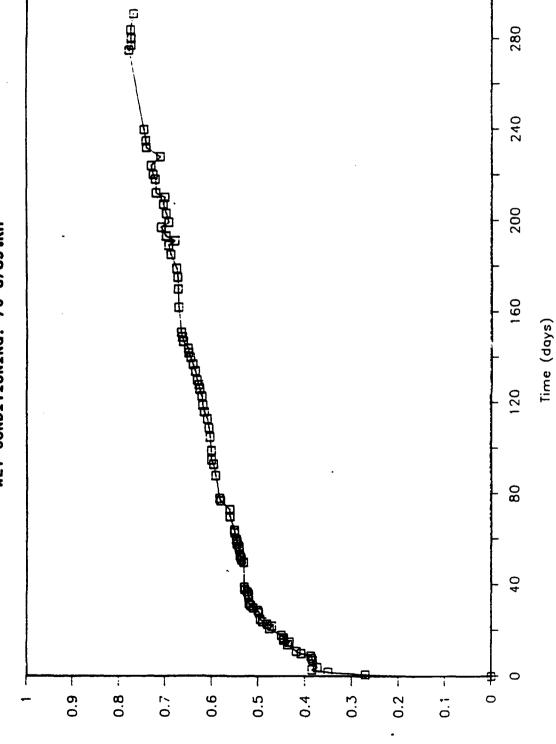
TESTED BY NUSIL TECHNOLOGY, IN ACCORDANCE WITH ASTM E-595, NASA SP-R-0022A, ESA PSS-01-702



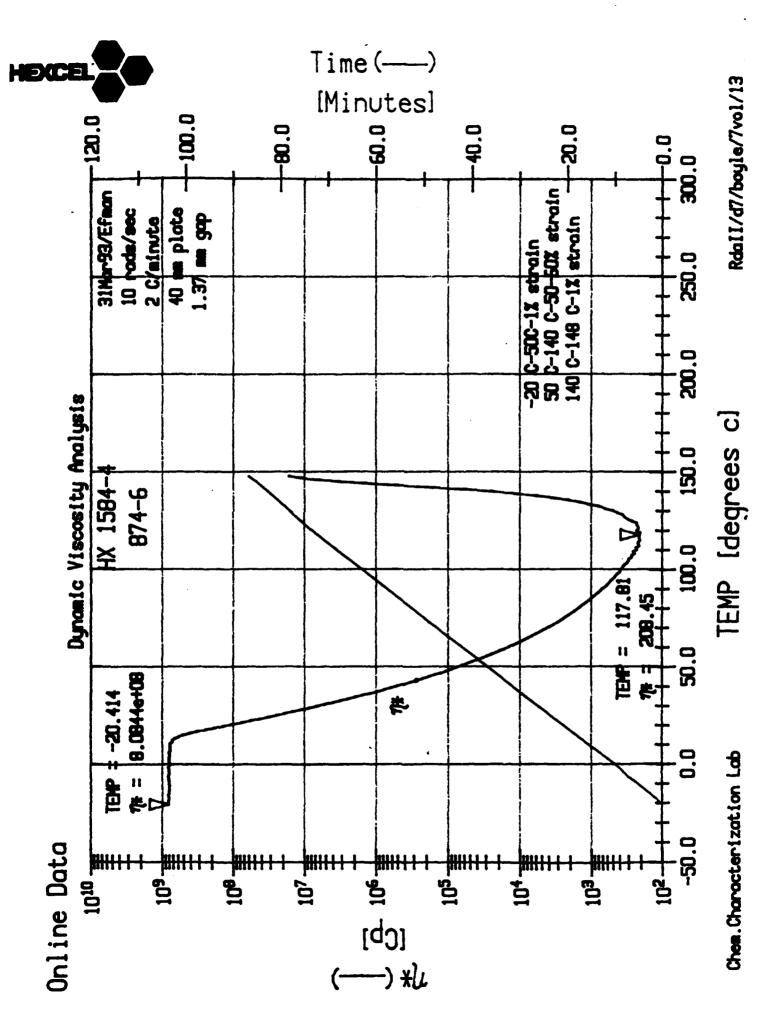


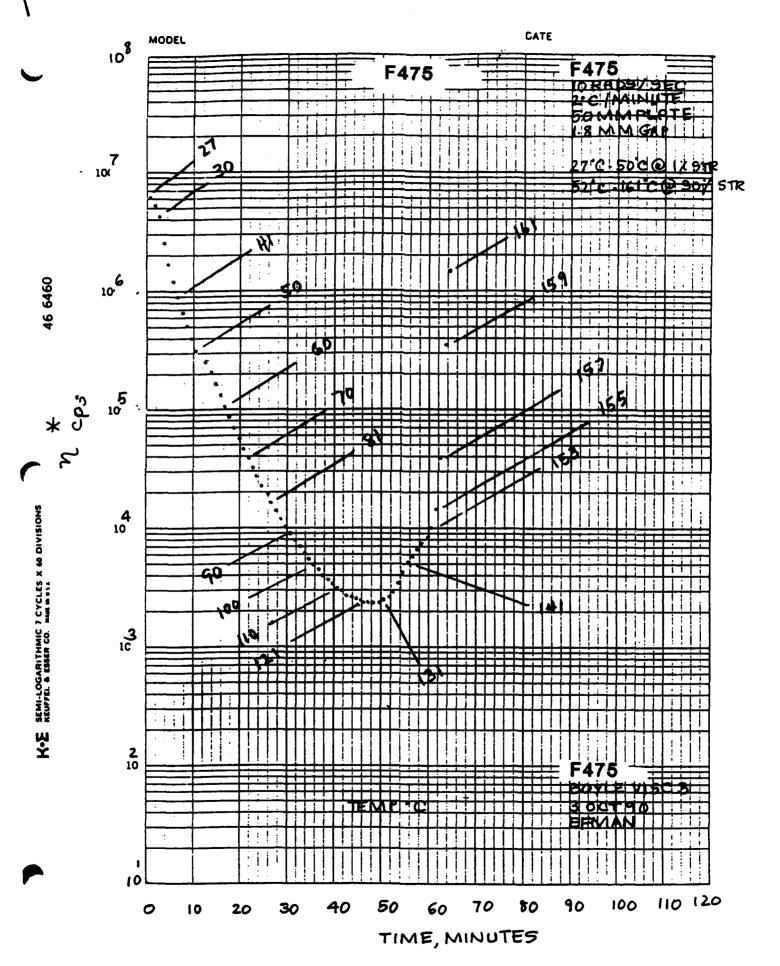


F475/T800, 0° LAMINATE WET CONDITIONING: 70°C/85\$RH



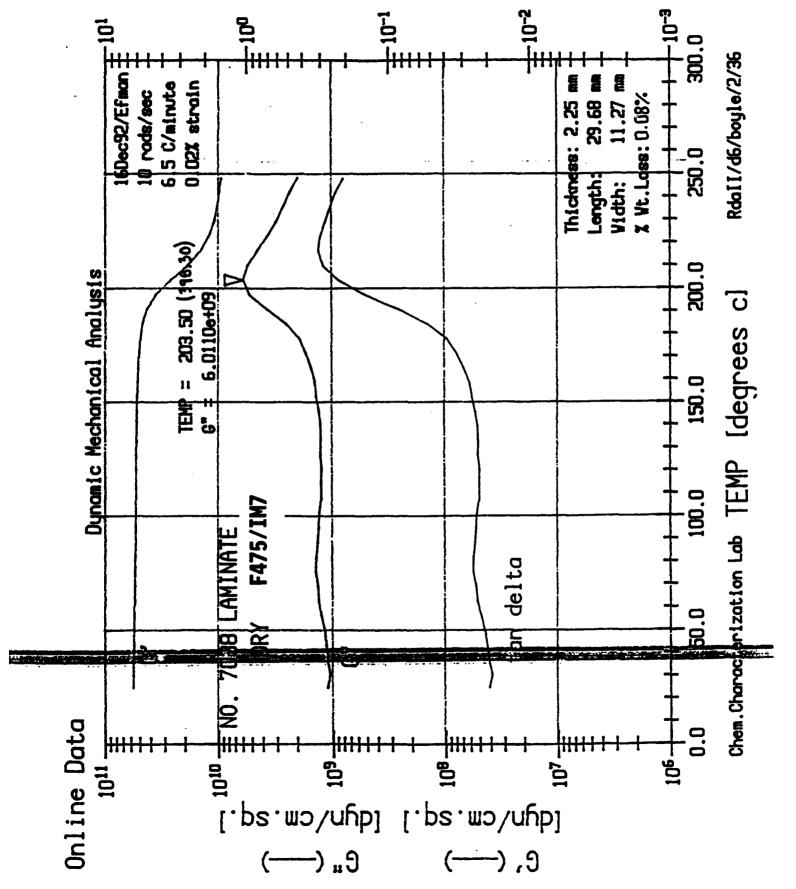
water absorption (%)

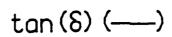




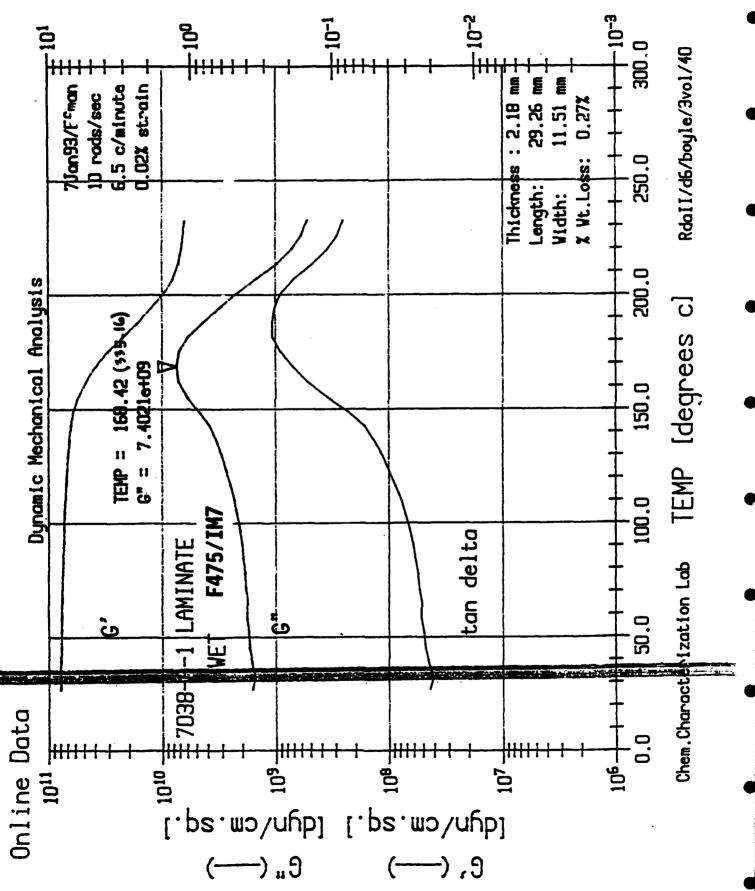


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### F475 MECHANICAL PROPERTIES T650-35 GRAPHITE FIBER, 12K

350°F DRY	\$ 1	:		i !		141	18.2		0.6
250°F WET	;	1	•	30.0		!			1
R.T. DRY	51.8	7.29	•	44.2		215	18.3		16.9
OPEN HOLE TENSION	STRENGTH (KSI)	MODULUS (MSI)	OPEN HOLE COMPRESSION	STRENGTH (KSI)	FLEXURE	STRENGTH (KSI)	MODULUS (MSI)	SHORT BEAM SHEAR	STRENGTH (KSI)

TENSION, COMPRESSION, AND FLEXURE VALUES ARE NORMALIZED TO 60% VF. SBS ACTUAL VF = 57.10% WET CONDITIONING, 144±8 HRS. IMMERSED IN 180 ±5°F WATER. NOTES:

HEXCEL PRODUCT NOMENCLATURE: 17G148-6-F475

### F475 MECHANICAL PROPERTIES M40J GRAPHITE FIBER, 6K

WARP TENSILE	ACTUAL DATA	NORMALIZED DATA
STR (KSI) MOD (MSI)	131 14.7	138
WARP COMPRESSIVE		

ACTUAL FIBER VOLUMES WERE 55.7% Data Normalized to 59% VF W4T386-39-F475

HEXCEL PRODUCT NOMENCLATURE:

STR (KSI) MOD (MSI)

### F475 MECHANICAL PROPERTIES M603 GRAPHITE FIBER, 12K

### R: T

303 46.0 6371	3.98 0.80 4879	105.3 45.3	38.3 0.98	9.70	7.40
0° TENSION STR (KSI) MOD (MSI) STN (με)	90° TENSION STR MOD STN	0° COMPRESSION STR MOD	90° COMPRESSION STR MOD	0° SHORT BEAM SHEAR STR	IN-PLANE SHEAR STR MOD

 $\mathcal{X}_{\varphi}$ 

ALL PROPERTIES EXCEPT SBS HAVE BEEN NORMALIZED TO A 59% VF. HEXCEL PRODUCT NOMENCLATURE: TSR 145-9-F475 NOTES:

### CHEMICAL STABILITY (TESTED BY SBS AT RT)

RETHANE, I HOUR	OPANOL, 1 HOUR 15.59	14.96		SBS STRENGTH (KSI) 14.96 15.59 15.70 15.40	CONDITIONING  DRY ISOPROPANOL, 1 HOUR  MEK, 1 HOUR  1,1,1, - TRICHLORETHANE, 1 HOUR  JP 4, 1000 HOURS
	RETHANE, 1 HOUR	DUR RETHANE, 1 HOUR	OUR RETHANE, 1 HOUR	15.05	SKYDROL, 1000 HOURS

TESTING PERFORMED ON IM7 REINFORCED LAMINATE. NOTE:

### DATA NOT INCLUDED. AVAILABLE

### **HX1566**

PRECISION SEGMENTED REFLECTOR PROGRAM REPORT (JPL). INCLUDES DATA ON THERMAL CYCLING, RADIATION EXPOSURE, MICROCRACKING, WATER ABSORPTION, AND MECHANICAL PROPERTY PERFORMANCE.

ELECTRICAL PERFORMANCE PROPERTIES NEAT RESIN

### HX1584-4

ELECTRICAL PERFORMANCE PROPERTIES NEAT RESIN ASTROQUARTZ 581 REINFORCEMENT SPECTRA 1000, 955 FABRIC REINFORCEMENT

MECHANICAL PERFORMANCE PROPERTIES ASTROQUARTZ 581 REINFORCEMENT ASTROQUARTZ 503 REINFORCEMENT SPECTRA 1000, 955 FABRIC REINFORCEMENT

CTE ASTROQUARTZ 503 REINFORCEMENT

### **F475**

ELECTRICAL PERFORMANCE PROPERTIES NEAT RESIN

MECHANICAL PERFORMANCE PROPERTIES GRAPHITE TAPE (IM7 FIBER)

**SMOKE DENSITY & GAS TOXICITY** 

### **CYANATE PREPREG COSTS**

### **GENERAL COMMENTS**

### **RESINS**

- CYANATE-ESTER RAW MATERIALS RANGE IN PRICE FROM \$15-100 PER POUND.
- TYPICAL FORMULATED PREPREG RESIN SYSTEMS RANGE FROM \$40-60 PER POUND.
- BLENDED OR MODIFIED SYSTEMS (I.E. ADDITION OF EPOXIES, THERMOPLASTICS, ETC.) WILL TEND TO BE SLIGHTLY CHEAPER.
- LONG TERM: PRICES EXPECTED TO DROP IF VOLUME USAGE MATERIALIZES.

### **PREPREGS**

- HIGH PERFORMANCE (HIGHER COST)
   CARBON FIBER REINFORCED PREPREGS
   TEND TO DILUTE THE EFFECTS OF
   CYANATE RESIN COSTS.
- COSTS CAN BE MODERATED BY:
  - LONGER PRODUCTION RUNS
  - TERM CONTRACT PRICING
  - REDUCED TESTING REQUIREMENTS

## HEXCEL SPACE RELATED ACTIVITIES

MEMBER OF HEXCEL IS AN INDUSTRIAL SPONSOR TO NASA CCDS (CWRU). ITAB.

COMPOSITE MATERIALS (P75/F584) FLOWN ABOARD STS-46 FOR EVALUATION OF A.O. RESISTANT COATINGS.

EVALUATIONS CURRENTLY BEING PLANNED WITH CYANATE MATERIALS FURTHER STUDIES (ACTIVELY MONITORED).

IRIDIUM SATELLITE CONSTELLATION WILL USE HEXCEL COMPOSITE MATERIALS FOR STRUCTURAL APPLICATION.

EOS (EARTH OBSERVING SATELLITE) DEM/VAL UTILIZING F475 COMPOSITE MATERIAL.

THAAD PROGRAM EVALUATING (M40J TAPE & FABRIC/F475) HEXCEL COMPOSITE MATERIALS FOR STRUCTURAL APPLICATIONS

**DEVELOPMENT WORK** 

HX1584-4, SOLUTION VERSION FILM ADHESIVE PRODUCTS SCREENING OF HX1584 WITH HM GRAPHITE FIBERS

### HEXCEL CORPORATION

# HEXCEL SUPPORTS GOVERNMENT CONTRACTING WITH ADVANCED QUALITY SYSTEMS

- LIVERMORE FACILITY OPERATING AT QUALITY LEVEL II SINCE 1988.
- LIVERMORE FACILITY COMPLIES WITH MIL-STD-1535A.
- MAJOR SUPPLIER TO USAF B2 PROGRAM.
- MAJOR SUPPLIER TO THE MILSTAR PROGRAM.

## FIBERITE-

# FIBERITE® 954 CYANATE ESTER SYSTEMS

## Graphite-Reinforced Polycyanate Composites **DoD/SDIO Workshop on**

June 16, 1993 Institute for Defense Analyses Alexandria, Virginia

2055 East Technology Circle, Tempe, AZ 85284 David B. Powell ICI Fiberite

# ICI Fiberite Global Facilities

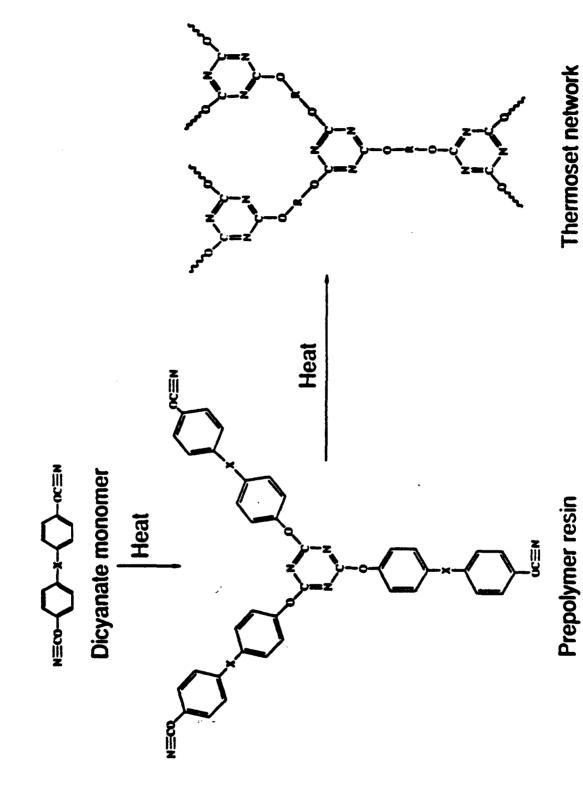
Manufacturing Sites	Floor Space (Sq. Feet)	No. of Employees	Products
Greenville, Texas	140,000	250	<ul><li>Tape</li><li>Fabrics</li><li>Weaving, Preforms</li></ul>
Orange, Calif.	130,000	145	<ul><li>Glass</li><li>Aramid</li><li>Thermoplastics</li><li>Ablatives</li><li>Tooling</li></ul>
Tempe, Ariz.	110,000	180	<ul><li>Tape</li><li>Fabrics</li><li>Roving</li></ul>
Winona, Minn.	300,000	285	<ul><li>Ablative</li><li>Molding Materials</li><li>Carbon/Carbon</li></ul>
Delano, Penna.	123,000	38	<ul> <li>Molding Materials</li> </ul>
Östringen, Germany	20,000	82	• Tape • Fabrics
TOTAL	853,000	086	G FIBERITE

# ICI FIBERITE SPACE MATERIALS BUSINESS

- Small segment of large prepreg-dedicated business
- Supply stability
- Profitable market segment (= survival)
- Tempe (Space Materials) site provides small scale flexible production capability
- Strong, effective relationships with fiber, raw material suppliers
  - to competently deal with application-specific requirements Technical capability (i.e. cl.-mistry, processing, testing) and raw materials
- Pricing based on value and technical content/support to provide cost-effective solutions
- Recent yearlong review of Fiberite space business reaffirmed commitment and focused business approach



# REPRESENTATIVE CURE CHEMISTRY OF DICYANATES



FIBERITE

# STRUCTURE-PROPERTY RELATIONSHIPS

### Structural Feature

### Performance Feature

Absence of H Bonding

- Low Moisture Absorption: 0.6 2.5%
  - Low Melt Point Monomers
- Balanced Dipoles (Short Moment)
- Low D<sub>k</sub>: 2.5 3.1

Aromatic Content

- Tg up to 650°F
- Inherent Flame Retardancy

- Relatively Low X-line Density (Gel at 60-65% Conversion)
- Toughness
- Tensile Elongation 2.5 6%
  - $G_{1c} = 0.8 1.2 \text{ in-lb/in}^2$





### 954 FAMILY

			PROCESSING CRITERIA
	FORMULATION	PERFORMANCE CRITERIA	MINIMUM VISCOSITY (cps)
954-2A	Second generation	Best balance of	>1500
	TP toughening; Co-continuous morphology	hot/wet performance and toughness	<2.5 mil tapes require high resin contents
954-3	"Pure" cyanate	Lowest hygrostrain	~100
			gravity and flexible caul flow problems possible



D93-022-5/0615

### A Data Sheet from



### 954-2A CYANATE RESIN

- Toughened cyanate using ICI Fiberite's proprietary thermoplastic technology
- High impact resistance
- 325°F (163°C) wet service temperature with post cure
- Attractive electrical properties
- 350°F (177°C) cure
- Controlled flow, good tack and out-life
- Available on a broad range of fibers and in forms including tape and fabric
- Autoclave or press-mold processable

Fiberite® 954-2A is a 350°F (177°C) curing toughened cyanate resin with a 325°F (163°C) wet service capability. Fiberite 954-2A is formulated for autoclave or press molding. Recommended cure is four hours at 350°F (177°C). Service temperatures are maximized by a post cure of 428°F - 450°F (220°C - 232°C). The recommended lay-up procedure for this material is L-7. The recommended cure procedure is C-33. The hold at 250°F (121°C) may be omitted, if desired.

Typical applications for 954-2A include primary and secondary aircraft structures, space structures, cryogenic tanks, or any application where impact resistance, light weight and excellent dielectric properties are required. Fiberite 954-2A can be impregnated via hot melt or solution techniques on all available fibers and fabrics.

### **TYPICAL NEAT RESIN PROPERTIES**

	RT	325°F	325°F/Wet (163°C/Wet)	350°F (177°C)
Tensile Strength, ksi	10.0	(103 0)	(103 C/Wel)	(177 6)
Tensile Modulus, Msi	0.44			
Tensile Ult. Strain, %	2.59			
Tensile Poisson's Ratio	0.38	:		
Tensile Strength, MPa	68.9			
Tensile Modulus, GPa	3.03			
Tensile Ult. Strain, %	2.59			
Tensile Poisson's Ratio	0.38			
Flexural Strength, ksi	16.9	13.5	11.9	12.6
Flexural Modulus, Msi	0.44	0.35	0.34	0.35
Flexural Strength, MPa	117	93.1	82.0	86.9
Flexural Modulus, GPa	3.03	2.41	2.34	2.41
H <sub>2</sub> O Uptake, wt. %	1.28			
Tg (DMA-Tan δ), °C	215, 249			
Density, g/cm <sup>3</sup>	1.24			

Notes: (1) Postcured 2 hours at 450°F

(2) Wet = Immersion at 160°F for 7 days

The data listed has been obtained from carefully controlled samples considered to be representative of the product described. Because the properties of this product can be significantly affected by the fabrication and testing techniques employed and since ICI Fiberite does not control the conditions under which its products are tested and used, ICI Fiberite cannot guarantee that the properties listed will be obtained with other processes and equipment.

### A Data Sheet from



### 954-3 CYANATE RESIN

- Low microcracking, low moisture absorbing cyanate resin
- -200°F to 250°F (-128°C to 121°C) service temperature without post-cure. After post-cure, service temperature is 325°F/wet.
- Attractive electrical properties
- 350°F (177°C) cure
- Very low minimum viscosity
- Available in a broad range of fibers and forms including tape and fabric
- Autoclave or press-mold processable

Fiberite® 954-3 is a 350°F (177°C) curing cyanate resin with a -200°F to 250°F (-128°C to 121°C) service temperature. Fiberite 954-3 is formulated for autoclave or press molding. Standard cure is two hours at 350°F (177°C). Service temperatures are maximized by a post-cure of 428-450°F (220-232°C). Fiberite 954-3 can be impregnated via hot melt or solution techniques on all available fibers and fabrics. The recommended lay-up procedure for this material is L-7. The recommended cure procedure is C-9.

Typical applications for 954-3 include primary and secondary space structures and other applications where resistance to microcracking and moisture, together with light weight and excellent dielectric properties are required.

### TYPICAL NEAT RESIN PROPERTIES

•	RT	325°F (163°C)	325°F/Wet (163°C/Wet)
Tensile Strength, ksi Tensile Modulus, Msi	8.2 0.4		
Tensile Ult. Strain, %	2.4		
Tensile Strength, MPa	56.5		
Tensile Modulus, GPa	2.76		
Tensile Ult. Strain, %	2.4		
Flexural Strength, ksi	17.3	12.6	11.2
Flexural Modulus, Msi	0.43	0.33	0.30
Flexural Strength, MPa	119.27	86.86	77.21
Flexural Modulus, GPa	2.96	2.28	2.07
H₂O Uptake, Wt. %	0.96		
Tg (DMA-Tan δ), °C Density, g/cm³	206 (N	PEST CUI	<b>4</b> 258 (w/PC)
Density, g/citr	1.19		

Notes: (1) Post-cured 2 hours at 450°F

(2) Wet = 7 day immersion at 160°F.

The data listed has been obtained from carefully controlled samples considered to be representative of the product described. Because the properties of this product can be significantly affected by the fabrication and testing techniques employed and since ICI Fiberite does not control the conditions under which its products are tested and used, ICI Fiberite cannot guarantee that the properties listed will be obtained with other processes and equipment.

### PITCH-BASED GRAPHITE FIBERS FOR SPACE APPLICATIONS

CLASS	TYPE (MFG'ER)	DENSITY Q/cm	TENSILE MODULUS Msi (GPa)	TENSILE STRENGTH ksi (GPa)	ELONGA- TION (%)	COMMENTS
<100 Msi Pitch	FT500MY, 1-3K (Tonen)	2.14	73 (505)	430 (3.0)	9.0	Strength, weaveability
(10µ fil. diam.)	P-75S, 0.5-2K (Amoco)	2.00	75 (520)	275 (1.9)	<b>6.4</b>	Database, US Domestic
,	XN50A, 0.5-2K (Nippon)	2.12	75 (520)	530 (3.7)	0.7	
	K135U, 0.5-4K (Mitsubishi)	2.12	90 (620)	450 (3.1)	9.0	High Strength
>100 Msi Pitch	FT700MY, 1-3K (Tonen)	2.16	105 (725)	470 (3.3)	0.5	Complex shape conformance
(10µ fil. diam.)	XN70A, 1-4K (Nippon)	2.16	105 (725)	530 (3.7)	0.5	•
•	P-100(S), 0.5-2K (Amoco)	2.14	110 (760)	350 (2.4)	0.3	US Domestic
	K139(U), 0.5-4K (Mitsubishi)	2.15	110 (760)	500 (3.4)	0.5	
	P-120S, 2K (Amoco)	2.18	120 (820)	275/325	0.22/0.26	US Domestic
	K13BU, 2K (Mitsubishi)	2.16	120 (830)	500 (3.4)	4.0	
	K1100X, 2K (Amoco)	2.20	140 (965)	450 (3.1)	. 2	K=>1000 W/m K

MANUFACTURERS ADVERTISED VALUES





## **HIGH MODULUS PAN-BASED GRAPHITE FIBERS** FOR SPACE APPLICATIONS

CLASS	TYPE (MFG'ER)	FIL. DIAM. microns	DENSITY g/cm	TENSILE MODULUS Msi (GPa)	TENSILE STRENGTH ksi (GPa)	ELONGA- TION (%)	COMMENTS
50-59 Msi	M35J 6,12K (Toray) T650/50X 12K (Amoco) M40J 6,12K (Toray)	5.5 5.1 5	1.75 1.71 17.1	50 (343) 52 (358) 55 (377)	683 (4.7) 650 (4.5) 640 (4.4)	1.3 1.2 2.5	Domestic
	1-50 3,6K (Amoco) M40 1,3,6,12K (Toray)	6.5	18.1	57 (393) 57 (393)	420 (2.9) 398 (2.7)	0.6 0.7	Domestic
60-69 Msi	M46J 6,12K (Toray) UHMS 3,12K (Hercules) M46 6K (Toray) M50J 6K (Toray)	5 7 5	1.84 1.88 1.88 1.88	63 (436) 64 (441) 65 (451) 69 (475)	611 (4.2) 550 (3.8) 370 (2.6) 569 (3.9)	1.0 0.8 0.6 0.8	Domestic
70-79 Msi	GY-70SE (Celion) M55J 6K (Toray)	8.4 5	1.96 1.91	<del>75 (517)</del> 78 (540)	270 (1.9) 583 (4.0)	0.4 0.8	Supply?- DISCONTINUED
80+ Msi	GY-80SE (Cellon) M60J 3,6K (Toray) M65J (Toray)	8.4 5	1.94	83 (572) 85 (588) 92 (634)	270 (1.9) 569 (3.9) 569 (3.9)	0.3 0.7 0.6	Supply? DISCONTINUED Experimental

Manufacturers' Advertised Values





## FIBERITE-

## **MATERIAL FORM FLEXIBILITY**

- 954 can be made in all product forms
- Reinforcements
- Quartz
- Glass
- Graphite
  - Kevlar
- Other

### Forms

- Fabric (up to 38" wide)
  - Tape (up to 24" wide)
- Tow (filament winding and tow placement)
  - Molding Compounds

### ICI / FCM Pilot Plant Tempe, AZ

### UNITAPE PRODUCTION CAPABILITIES

- ▼ Fiber Areal Weight 30 gm/m<sup>2</sup> to 380 gm/m<sup>2</sup>
- ▼ Resin Areal Weight 21 gm/m<sup>2</sup> to 400 gm/m<sup>2</sup>
- ▼ Tape Width 1" to 24" wide
- ▼ Able to use several different papers.
- ▼ Able to film highly filled resin systems.
- ▼ Capable of filming pseudo hot melts.

NOTE: Low FAWs require generally higher Resin

Content ranges, along with small tow sizes.

200591-2/123/IB lmjr:200591 rev:240792

### ICI/FCM Pilot Plant Tempe, AZ

### SOLUTION PREPREGGING

- Three-zone, 36 inches wide with temperature capabilities up to 600°F
  - Fabrics up to 34" wide
  - FX products (4 tows)
  - String molding compounds
  - Mat products up to 34" wide
- Drum Winder
  - 12" wide max width
  - 72" length

270792-1/123/IC | lmjr:270792

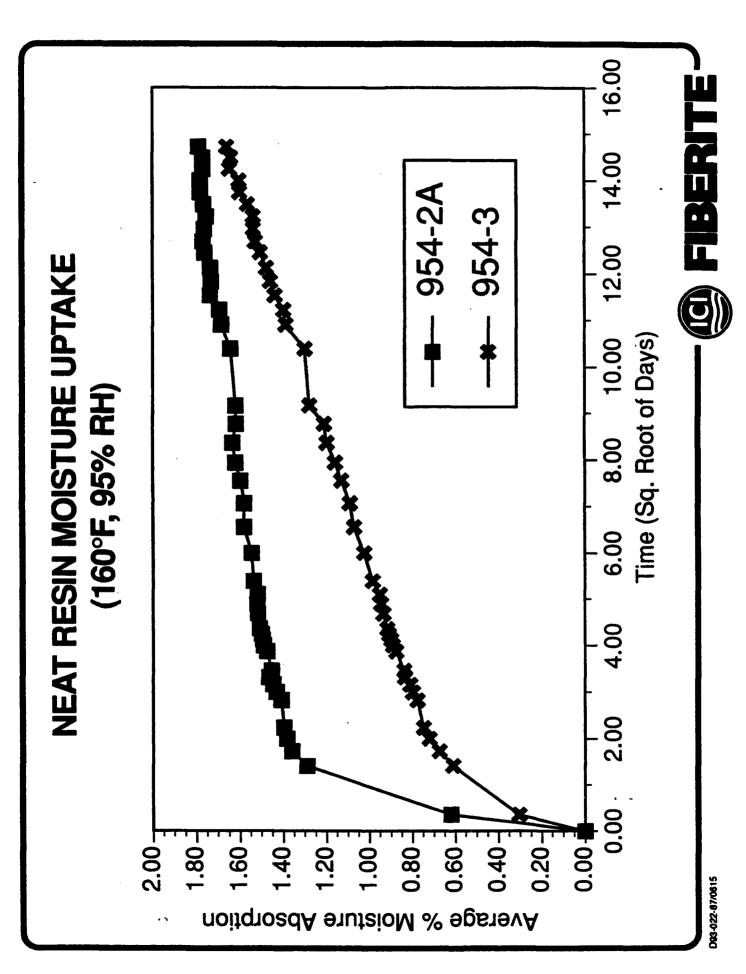
# ICI FIBERITE CYANATE SYSTEMS MATURITY

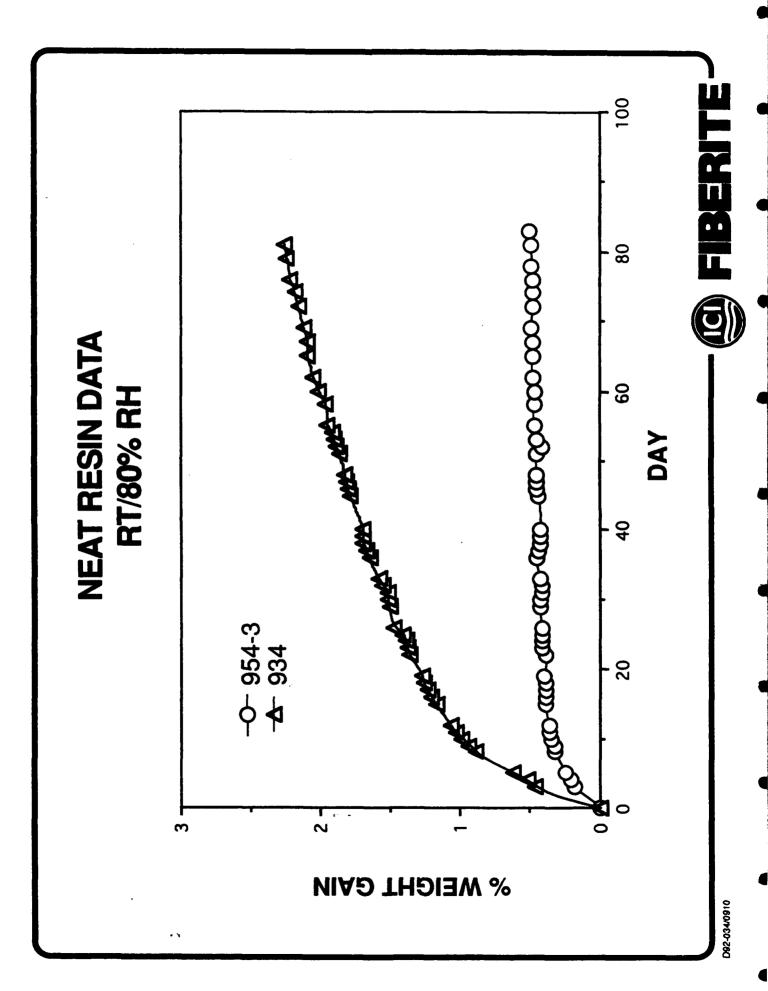
Active R&D on cyanates at ICI Fiberite since 1986

	954-2/-2A	954-3	954-1 (being phased-out)
First Production Quantities	Q1/1989	Q2/1989	Q2/1987
Total Prepreg Production (lbs)	> 3,500	> 6,500	4,000
Approx. No. of Lots	80	90	30
Major Programs	Milstar E-4 Radomes Commercial Satellites SSC Detector AXAF-I "Others"	AXAF-I MSS "Others"	Milstar Black Radomes MEC Solar Arrays



**BERITE** 





## DIMENSIONAL STABILITY

108.5

29.2

18.9

0.704

0.174

 $0.18^{3}$ 

Hygrostrain, ppm

Water Absorption, %

CME, ppm/%

LAYUP

(O, 60,120)xs 1.e. 0°=1/3

172 105

155

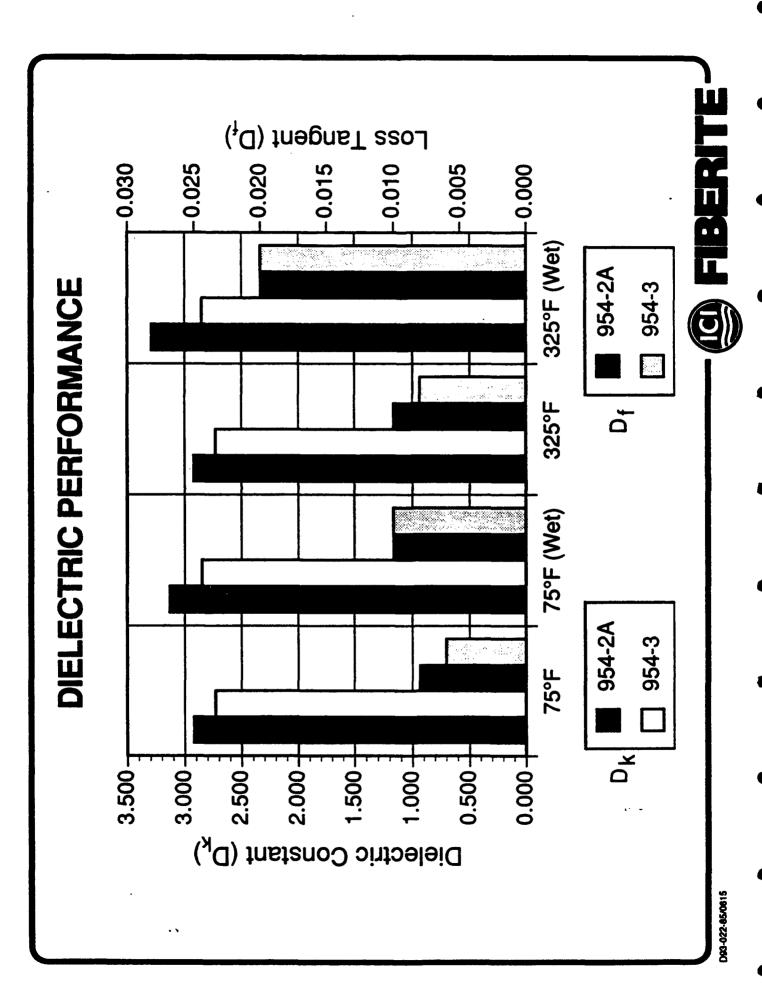
(0'30'00'40'120'120)2

Psuedo isotropic P75 laminates; 30% RC

Hygrostrain divided by %M = CME

R. Brand and E. Derby; SPIE Conf, 1690, 309. April '92 (Composite Optics, Inc.) C. Blair and J. Zakrzewski, SPIE Conf, 1690, 300. April '92 (Lockheed MSC) 55% R.H/EQ 50% R.H/EQ





## 954 NEAT RESIN ELECTRICALS

_		954-1	954-2A	954-3
75°F	ð t	2.88	2.92	2.73
325°F	ăă	1 1	2.92	2.73
75°F/Wet	ă ă	3.08 0.02	3.13	2.85
325°F/Wet	ăă	, ,	3.29	2.85
% H <sub>2</sub> O		1.94	1.69	1.29

- Tested per ASTM 2520D at 10.0 GHz
- Wet = 160°F, 95% R.H. for 140 days
- Hot/Wet samples measured after 15 minutes at 325°F with no change in weight to nearest 0.01g (sample weight 2.7g) i.e. < 0.4% drying
  - All samples cured 2 hours @ 350°F; post-cured for 2 hours @ 428°F





# 954-3 OUTGASSING/CONTAMINATION

	P-75/954-3 LAMINATE(1)	954-3 NEAT RESIN <sup>(2)</sup>	T-300/934 LAMINATE <sup>(2)</sup>	NASA MSFC 1443 LIMITS
Total Mass Loss (TML)	0.09%	0.20%	0.4%	< 1.0%
Collected Volatile Condensable Material (CVCM)	< 0.01%	< 0.01%	< 0.01%	≤ 0.1%
Water Vapor Regained (WVR)	0.06%	0.04%	0.13%	
Optical Witness Sample (OWS)Maximum AR/R (121.67-200nm)	- 1.7% (160 nm)		No Change	- 3.0%

(1) NASA MSFC data (5/20/93), Spec 1443, 60°C sample, 20°C OWS/Collector, 10-6 Torr, for AXAF-I Program.

(2) Tested per ASTM E595.





### **MICROCRACKING**

		CRAC	CRACKS/INCH (AT NO. OF CYCLES)	TNO. OF CY	(CLES)
RESIN	PLY THICKNESS (MILS)	0	10	20	91
934	5.0	4/2	17/49	45/62	47/65
954-3	2.0	1/0	5/3	8/2	14/6
934	2.5	0.0	1/3	1/4	1/5
954-3	2.5	0/0	0/0	0/0	0/0

- P-75 laminates, (45, 0, -45, 90)<sub>3s</sub>, 60% FV, 2 in. x 2 in.
   Cycle: -150°F to +150°F and back @ 20°F/min., 5 min. dwell at extremes
   Data taken at 50x magnification.
   0° normal edne/90° normal and pack at the context of the co



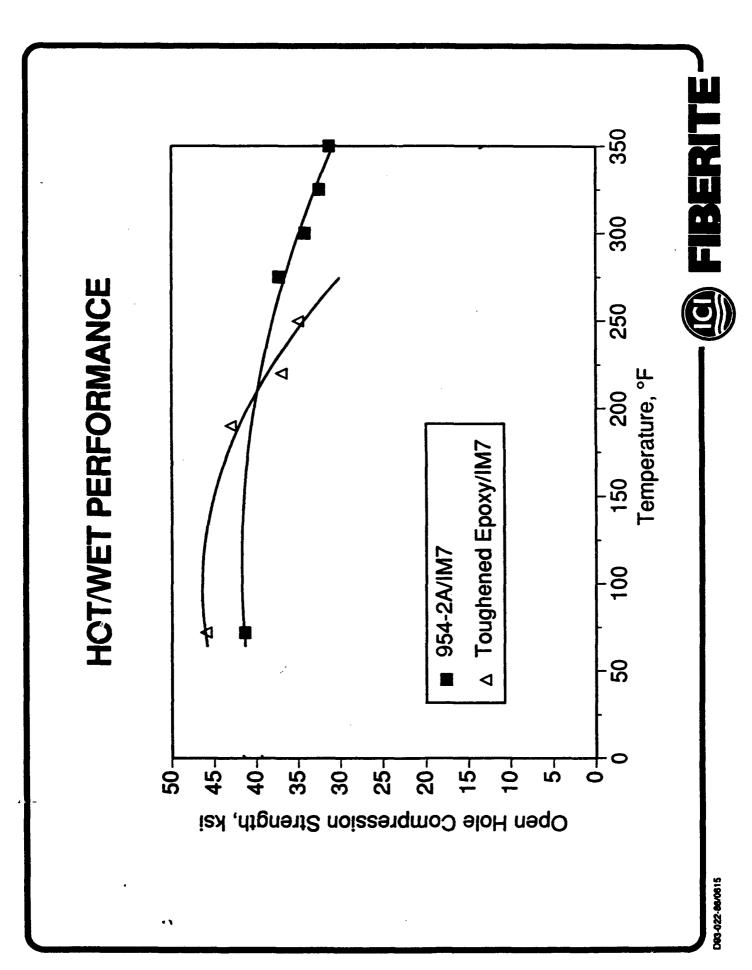
FIBERITE-

## FIBERITE-

Dg3-022-89/0615

## **OXYGEN PLASMA STABILITY**

Resin System	Mass Loss Factor (μg/cm² hr.)
934 epoxy	701
954 cyanates	280-600



## Room Temp Testing, ±45 Tensile Coupons SOLVENT SENSITIVITY OF HY-E 1354-2AT

-	Control	Water	JP-4	Skydrol	MEK	IPA
±45 Tensile Strength, ksi	34.0	33.4	1	30.7	31.6	34.6
±45 Tensile Modulus, Msi	2.24	2.25	I	2.05	1.96	2.32
Shear Strength, ksi	17.0	16.7	1	15.3	15.8	17.3
Shear Modulus, Msi	0.62	0.62	ı	0.56	0.53	0.64
Poisson's Ratio	0.80	0.81	ı	0.83	0.83	0.81
Shear Modulus Retention, %	×	100	<b>I</b>	06	82	103
Conditioning Temperature, °F	×	150	HT	180	H	Ħ
Wt. Gain, %	×	0.43 0.43	ı	0.58	0.6 7 days	0.07
		١٠		ov vays	/ days	ov days

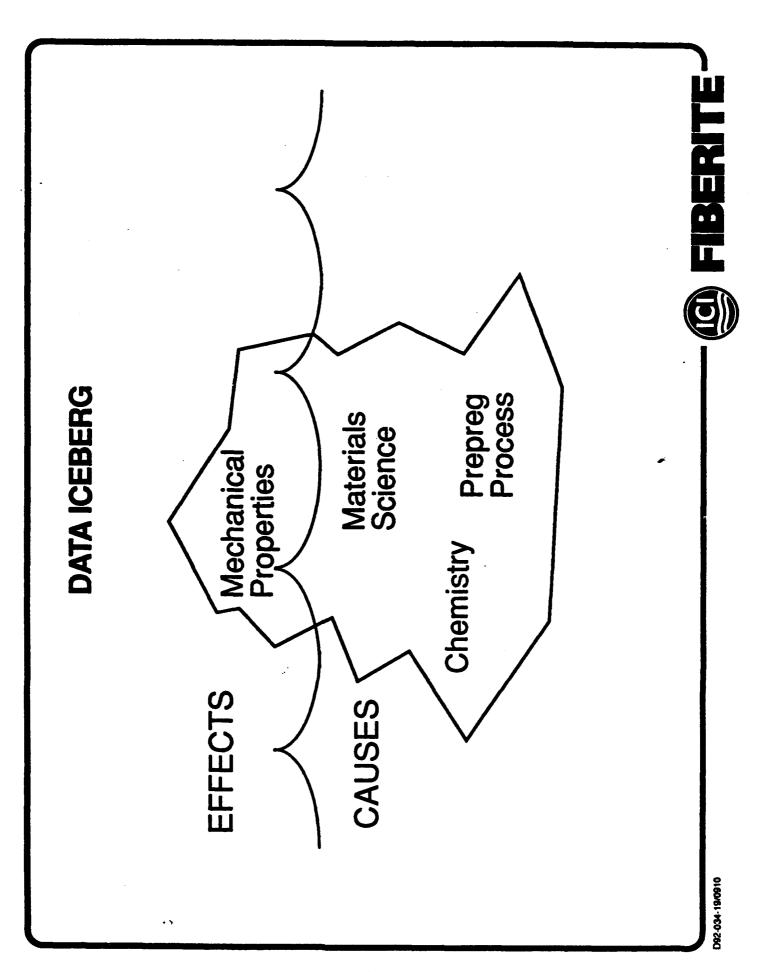
Data not shown is in testing (9/10/92)

4 ply ±45 tension samples tested at RT after indicated exposure 13(54-2A)T, Lot 26040

Cure: 4 hours @ 350°F, 2 hours @ 450°F







### FIBERITE Temperature Pressure Line Speed Etc. PROCESS VARIABLES 954-2A ICI FIBERITE DATABASE 977-3 MATERIAL SCIENCE VARIABLES ICI FIBERITE Strength Modulus **DATABASE** FIBER Form Sizing 977-2 977-1 RESIN VARIABLES Epoxy A Epoxy B Hardener TP<sub>x,yz,n</sub> 1397 IN4/INSES

## FIBERITE

## WHY CO-CONTINUOUS?

- Optimal Mechanical Properties (toughness vs. compression)
- Scale of Morphology/Product Forms
- Morphological "Stability"
- Excellent Solvent Resistance, Creep
- Excellent Mechanical Outlife
- Robust Cure Cycle

### 954 PREPREG

Data Category	Pro	Property	Comments
	General	Specific	
Prepregging	Scale-Up	Fiber Types	Glass, Quartz, Pitch, High and Intermediate Modulus PAN
		Process Parameters	Line Speed, Temperatures, Pressures
		Prepreg Measure- ables	Flow, Tack, Drape, Gel, HPLC, IR, Handleability (Soft Issues), FAW, RC, Vols.
		Product Capability	FAW and RC Limits, Tow Sizes, Equipment





## FIBERITE-

## 954 CHARACTERIZATION

	954-1	954-2/-2A	954-3
IM PAN	S		()
PITCH	S	/	1
GLASS	•		ı
QUARTZ	•	2	[2]

HAVE DATA
IN TESTING
CUSTOMER PROPRIETARY DATA

D92-034-20/0910

# 954 LAMINATE DATABASE OVERVIEW

Data Category	Prop	Property	Comments
	General	Specific	
Materials Characterization	Tension	0°, Warp	RT: Strength, Modulus, Strain
·		°06	RT: Strength, Modulus, Strain
		With Holes	Open
			25/50/25 Layup, RT.
		,	180-300°F Wet: Strength, Modulus



FIBERITE-

# 954 LAMINATE DATABASE OVERVIEW

Data Category	Property	erty	Comments
	General	Specific	
Materials Characterization	Compression	0°, Warp	RT - 300°F, Dry, Wet, Strength, Modulus
		With Holes	Open
			25/50/25 Layup, RT, 180°F to 350°F Wet, Strength, Modulus
	-	After Impact	270 in-lb, 25/50/25 Layup
	Shear	0°, Warp	ILSS, RT to 350°F, Dry, Wet
		00	IPS, Strength, Modulus, Poisson's, RT to 325°F, Wet



D93-022-67/0615

# 954 LAMINATE DATABASE OVERVIEW

Data Category	Property	erty	Comments
	General	Specific	
Materials Characterization	Flex	0°, Warp	Strength, Modulus, RT
		06،	Strength, Modulus, RT
******	Fracture	, G	DCB
	Toughness	G <sub>lic</sub>	ENF
	Edge Delam.	Strength	Onset, Ultimate, RT
	Thermal Cycling Microcrack	Photo- micrographs	-65°F to 310°F 100 cycles -150°F to 150°F 100 cycles
· -	Solvent	S <u>d</u>	JP4, Skydrol, IPA
	Besistance	:	
		±45 Tension	Solvents: JP-4, Skydrol, MEK





## Properties Summary for Various 70-90 Msi Pitch Fibers in Fiberite 954-3 Cyanate Resin.

Material Code Hv.F.	2054-3D	4054-3D	4954-3A	5054-3A
Fiber Manufacturer	AMOCO	TONEN	MITSUBISHI	NIPPON
Fiber Designation	P-75S 2K	H	K-135 2K U	XN-50A 24
	, , ,			
Fiber Tensile Modulus, (Msi)	75	2	8	75
Fiber Tensile Strength, (ksi)	275	430	200	230
Fiber Elongation, (%)	0.4	9.0	0.4	9.0

Laminate Properties	2011				
0° Tensile	Strength, (ksi)	157	250	290	250
	Modulus, (Msi)	43	42	22	£3
	Strain, (%)		0.58	0.53	
90° Tensile	Strength, (ksi)		4.3	3.9	5.3
	Modulus, (Msi)		0.93	0.82	0.73
	Strain, (%)		0.46	r 49	
0°Compressive	Strength, (ksi)	62	70.	29	63
•	Modulus, (Msi)	35	30.	40.	35
0° Flexural	Strength, (ksi)		110	66	100
	Modulus, (Msi)		30.	42	33
90° Flexural	Strength, (ksi)		9	8.8	
	Modulus, (Msi)		0.00	0.80	
0° SB Shear	Strength, (ksi)	8.2	10.	11	11

### Notes:

- 1. These data from various sources with varying numbers of tests. The data should be
- taken as a general indication of the materials' capabilities only. All data are normalized or nominalized to 60% fiber volume fraction except strain and short beam shear strength. જાં



# Comparison of Fiber and Composite Properties for GY-70 and Replacement Candidates in Fiberite 934 and 954 Resins

Material Code. Hv-E:	نن	1534A	3434J	3454-2AH	2034D	5054-3C
Fiber Manufacture		CELION	TORAY	TORAY	AMOCO	NODAIN
Fiber Designation		GY-70 SE	M55J 6K	M60J 6K	P-75 2K	XN50A 0.5K
Fiber Tensile Modulus, (Msi)	ulus, (Msi)	75	28	82	75	75
Fiber Tensile Strength, (ksi)	nath, (ksi)	270	583	269	275	530
Fiber Elongation, (%)	%)	0.36	0.8	0.7	0.4	0.7
Laminate Properties	les					
0° Tensile	Strength, (ksi)	100-115	272	316	141	230
	Modulus, (Msi)	42-46	84	23	45	47.1
	Strain, (%)	.2425	0.54			
90° Tensile	Strength, (ksi)		4.16			9
	Modulus, (Msi)		0.91			0.81
	Strain, (%)		0.45			
0°Compressive	Strength, (ksi)	55-70	117.	127.	61.	61.
	Modulus, (Msi)	35-38	44.	48.	36.	44.
0° Flexural	Strength, (ksi)	110-125	147	162	118	117
	Modulus, (Msi)	36-40	42.	45.	35.	35.
90° Flexural	Strength, (ksi)					
	Modulus, (Msi)					
0° SB Shear	Strength, (ksi)	9-11	8.5	10.0	8.5	11.7

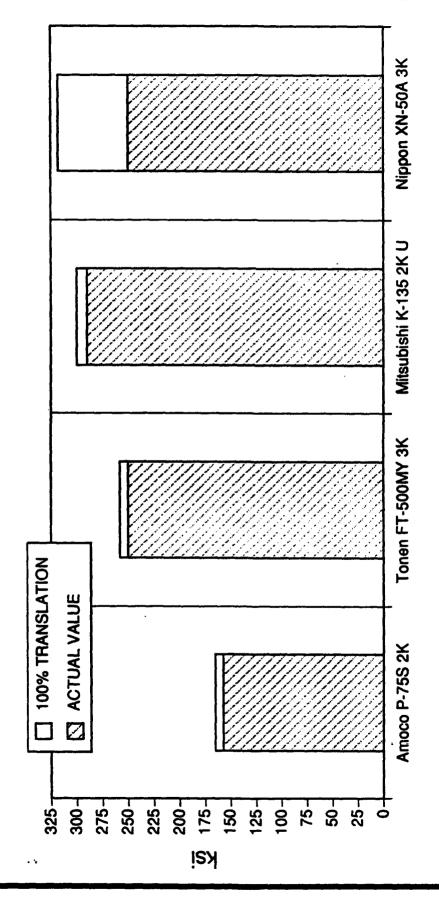
### Notes:

- 1. GY-70 data from Materials Handbook. M55J data from DOC 34-134 (15 tests ea). P-75 data from MTDB capability study. Others from certification data.
  - 2. All data are normalized to 60% fiber volume fraction except strain and short beam shear strength.





## **TENSILE STRENGTHS OF 70-90 Msi PITCH FIBERS IN FIBERITE 954-3**



### NOTES

- 1. These data from various sources with varying numbers of tests. The data should be taken as a general indication of the materials' capabilities only.
- All data are normalized to 60% fiber volume fraction except strain and short beam shear strength.





## **BONDING OF CYANATE LAMINATES**

Material

Double Lap Shear Strength

20(34)D

 $2776 \pm 128$ 

20(54-3)D

 $2924 \pm 85$ 

20(54-1)D

3968 ± 83

per ASTM D3528

Hysol EA 9394 Adhesive



## **EPOXY LIKE PROCESSING**

- Autoclave, Press-mold, Vacuum Bag
- 350°F Cure temperature, Optional Post Cure
- Flexible
- Bagging, and Bleeding Schemes as for Epoxies

## 954 MANUFACTURING

	PROF	PROPERTY	
DATA CATEGORY	GENERAL	SPECIFIC	COMMENTS
Manufacturing (Parts)	Cure Flexibility	Neat Resin	Tg, H <sub>2</sub> O Absorption, Compression Strength, Flexure Strength, Modulus, G <sub>ic</sub> , K <sub>ic</sub> , Density, Morphology, Cure and Post-Cure Study (2 - 18 hrs./350°F)
		CAI, OHC	Straight up vs. hold at 250°F
<b>(</b> * -		Тд	Flex (Dry), DMA, G', G", Tan ∆, Dry, Wet



D83-022-70/0615

## 954 MANUFACTURING

. `	PROF	PROPERTY	
DATA CATEGORY	GENERAL	SPECIFIC	COMMENTS
Manufacturing (Parts)	Out-Time	Morphology	0-42 Days; Phase Description
-		HPLC	0-42 Days; Peak Ratios
•		SC	0-42 Days; ΔH; Onset and Peak
			Temperature
		Rheometrics	0-42 Days; Viscosity
		Flow	0-42 Days
		Gel	0-42 Days
		Tack	Qualitative
		SBS	RT, 190°F Wet, 250°F Wet
	Thick	MED Panels	Ply Dropoffs: 2'x3' panels
	Sections	& Thick Parts	2'x2', >1.0" thick: Tg & DSC study
		Customer	
		Evaluațions	





## 954-2A NEAT RESIN OUTLIFE STUDY

	DAY 0	<b>DAY 14</b>	DAY 28	DAY 42
DSC peak (°C) onset (°C)	255 211		253 180	249
n 88°C 30' (cps)	12-13K	14-15K	20-22K	30-33K
M min. (cps)	1050	1300	2200	2300
Morphology	Co-Continuous		Co-Continuous	
Tack/Drape	y/y	y/y	y/y	y/y

Resin was stored at 0°F until 7/15/92 and then sampled and aged at room temp Cure: 4 hours/350°F, 2 hours 450°F



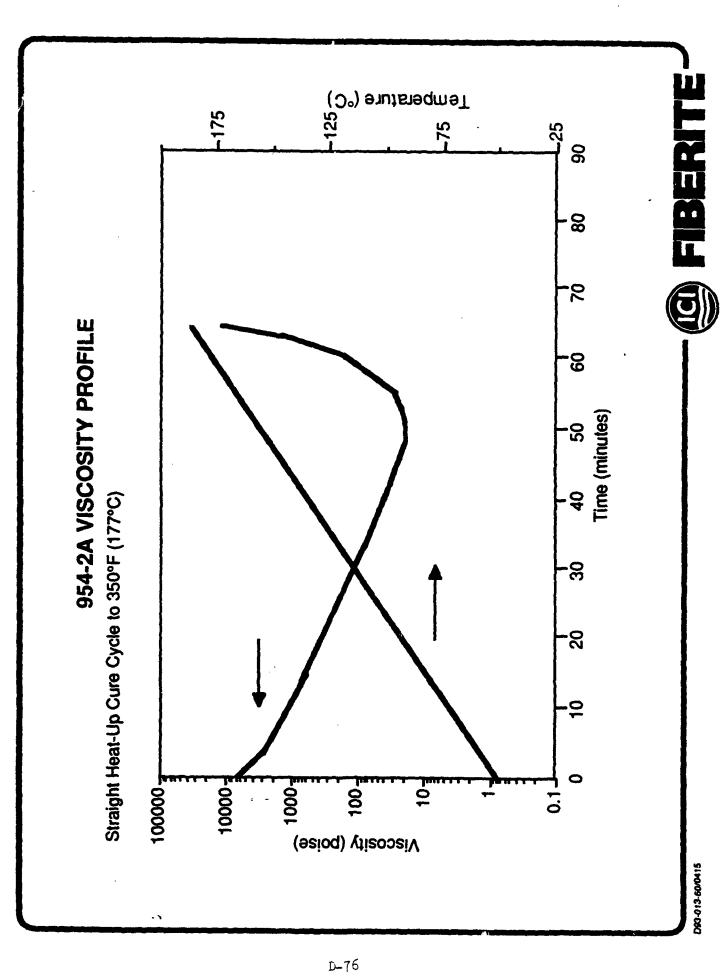
## 954-2A PREPREG OUTLIFE STUDY

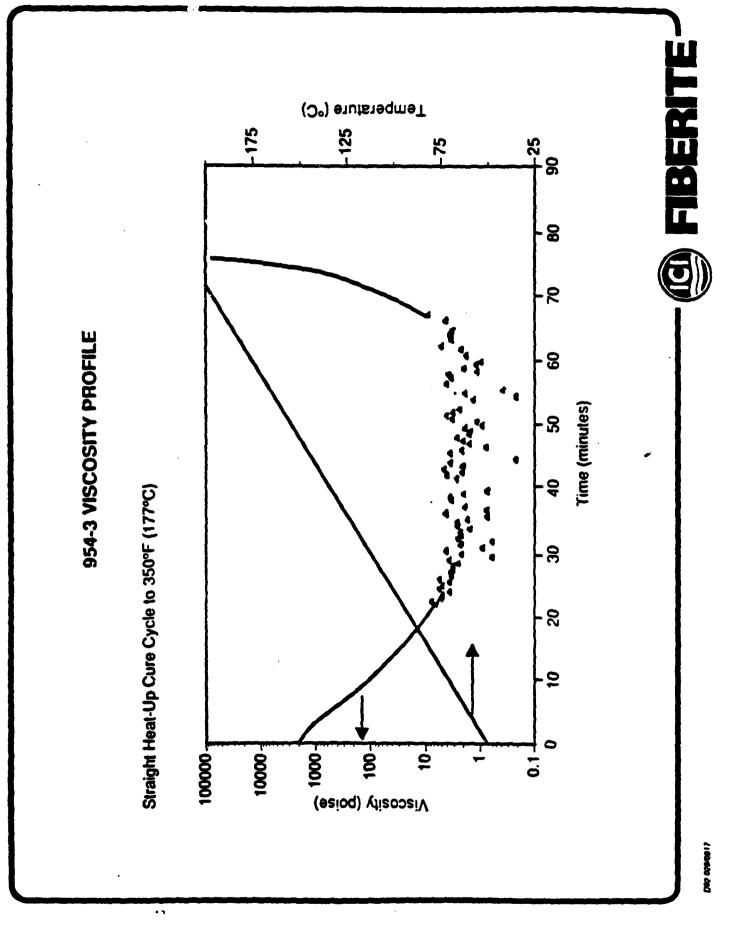
	DAY 1	<b>DAY 14</b>	DAY 28	DAY 42
Flow %	15.48	14.87	13.44	12.70
DSC peak (°C) onset (°C)	212 155	213 168	214 165	205 156
Tack/Drape	٨/٧	λ/y	λ/υ	lu/y

Prepreg 13(54-2A)T, Lot #26032, RC = 35±3%, FAW = 145±5g/m² Samples cut and stored in sealed bag at RT until tested.



D92 034 11/0910





### PERFORMANCE VERSUS COST (\$ ber bound)

FIBER	T-300 6K 33 Msi	P-75 2K R 75 Msi 8	M60J 85 Msi 6K 3	Ä	P-120 2K 120 Msi 640 W/m°K
954-2A, -3 (Cyanate Ester)	175	550	029	066	1450
934 (Industry Standard Epoxy)	140	515	635	955	1415
Price Delta for Cyanate Ester	25%	%2	2%	4%	2%

- Not a price quotation. Numbers are ROM for comparison purposes only.
- Actual pricing based on customer-specific requirements is available from Mary Gardea (Tempe, AZ) at (602) 730-2166.



FIBERITE.

### POTENTIAL RISKS

MATERIAL-RELATED RISK (Consequence)	CURRENT	TREND
<ul> <li>Mechanical, Thermal Failure (System Failure)</li> </ul>	Very Low	Decrease (Inverse to Database)
Processing Failures     (Program Cost, Schedule)	Low	Decrease (Inverse to Experience Base)
Supply Stability - Raw Materials     (Program Schedule, Requal, Cost)	Moderate	Decrease (Inverse to Market Size)
<ul> <li>Supply Stability - Prepregger Demise (Program Schedule, Requal, Design Options)</li> </ul>	Company & Overall Market Dependent	Increasing



# INPUTS FOR MATERIALS DEVELOPMENT

### DESIGNER CONCERNS

- Mechanical Performance
- Thermal Performance
  - Dimensional Stability
- **Outgassing Performance**
- Service Temperature (Tg)
  - **Cured Ply Thickness**
- Self-adhesive Capability

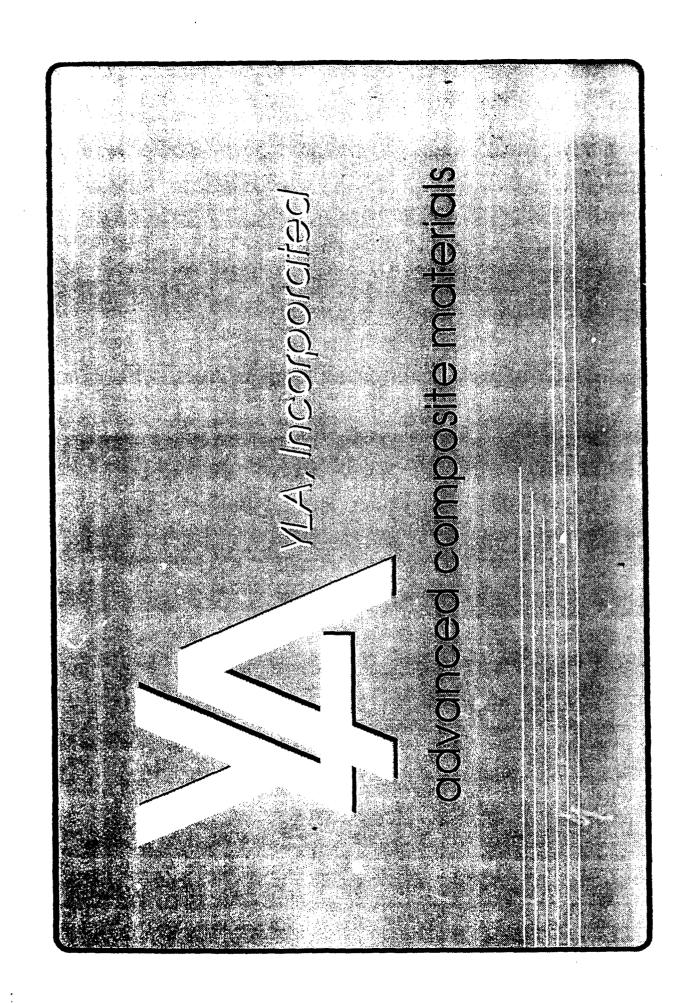
## PREPREGGER CONCERNS

- Raw Material Reactivity
- Supply Stability
- Mixed Resin Viscosity and Reactivity
- Pot Life/Shelf Life
- Fiber Wet Out
- Sizing Compatibility
- Environmental Issues

### **FABRICATOR CONCERNS**

- Cure Cycles
- Flow Control
- Bleed Scheme
- Resin Content/FAW control
- Outlife
- Tack/Tack Life
- Residual Stress
- Tool Release
- Bondability/Repair
- Drape/Radius Conformance
  - Environmental Issues





### **YLA** As of June 16, 1993

Employees: 15

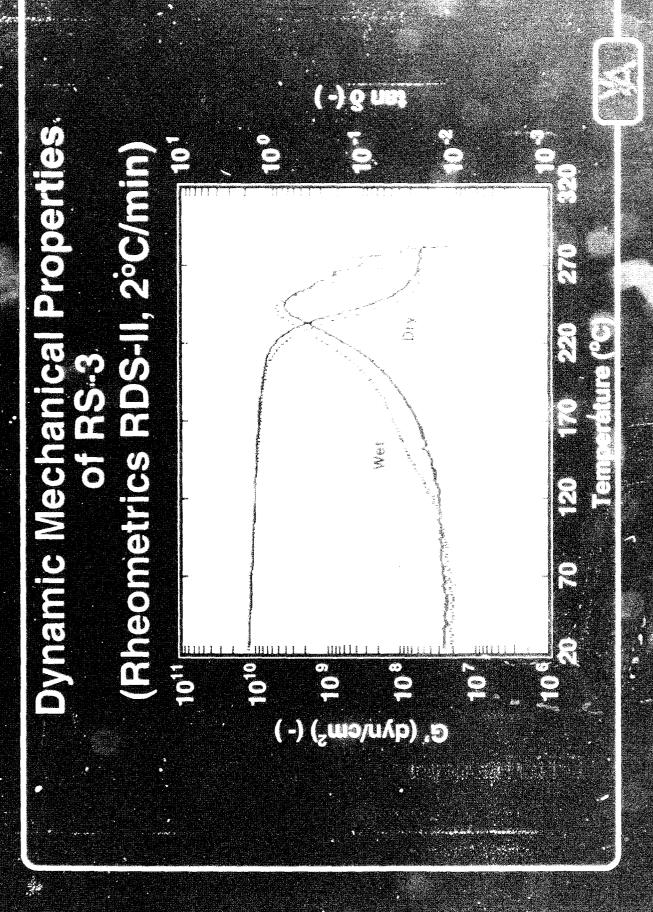
54" wide hot melt fabric impregnator & filmer 12" wide unidirectional tapes Material Processing Capability: 6 station prepreg tow

Move into larger facility site in Benicia - Jan, 1994 Select European manufacturing site - Oct, 1993 Submit for registration to ISO 9001 - Dec, 1993

Goals for 1993;

### **RS-3 History**

- Feb, April 1988 RS-3 developed. Initial development was directed toward Boeing's SRAM II.
- Aug, 1988 RS-3 submitted to Loral Space Systems.
- July 1989 RS-3 technology licensed to Nippon Petrochemical
- Dec, 1989 Loral Space Systems Specification, A020281, prepared around RS-3.
- Jan, 1990 Qualified at TRW. -3 specifications.
- Jan, 1990 RS-3 evaluation started at Mc Donnell Douglas Technologies.
- Nov, 1992 RS-3(M)/SF-5 Microply F-15 radome flight article built by McDonnell Douglas Technologies.
- Jan, 1993 RS-3 Qualified to COI's Spec CMS 002
- Feb, 1993 RS-3 Qualified EDO's Spec. EMPS 1046.
- Feb, 1993 RS-3 Qualified to Loral Spec. A020300.



## **RS-3 RADIATION RESISTANCE**

## TESTING PERFORMED BY JPL

modulus of RS-3 was then determined. Flexural strength producing 1 Mev electrons. The change in flexural and modulus of RS-3 after 109 rads shows virtually 10<sup>7</sup>and 10<sup>9</sup>rads in JPL's Dynamitron accelerator, "Neat resin samples were exposed to doses of no change."

Flexural Modulus, Msi

10<sup>9</sup> rads 107 rads Central

.47

.48

53

# RS-3 OUTGASSING

TESTING PERFORMED BY SPACE SYSTEMS LORAL

VCM .000. TML .094% .071 .130 .115

AVG .102%

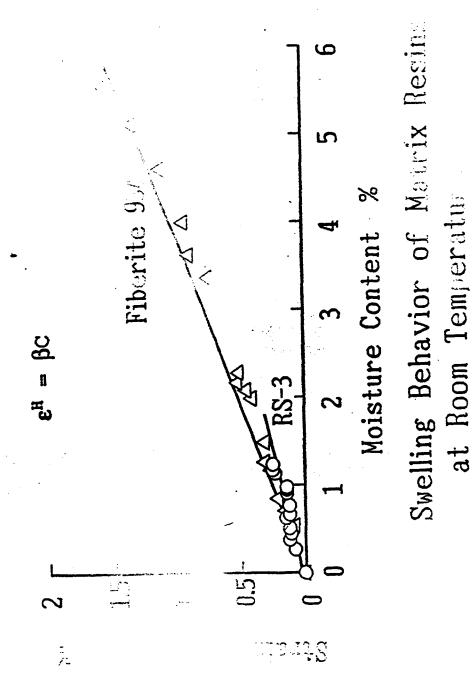
000

TESTING PERFORMED BY BOEING DEFENSE AND SPACE

TML .084% VCM .001%

sture Absorbing Properties of Matrix Resi

	Moisture Diffusivity K <sup>H</sup> m <sup>2</sup> /s	Max. Moist Content Cm %	Max. Moisture Content Cm %	Coefficient of Moisture Expan	Coefficient of Moisture Expansion & m/m
	Room Temp. 93°C	<b>R.</b> T.	93.C	e e	93.0
RS-3	8 0 10 -11 8 11 84 x 10 -11	1.2	1.5	0.149	0.156
F934	8.31x10 <sup>-14</sup> 2.69x10 <sup>-12</sup>	9	7	0. 261	0. 237



# RS-3 Prepregging - Space Structures

### Fibers:

XN-50, XN-70, XN-80 P75, P100, P120

K139

M46J, M55J, M60J

MHO

Kevlar

### Unidirectional Tapes:

Lbs. - Approximate FAW - g/m² 27 32 70-80 150

160 500 300

Fabrics:

92 lbs XN-50 .5K 24x24 5HS-42"

XN-50 1K 12x12 Plain-16" XN-50 1K 16x16 4HS24"

Kevlar 120-38"

60 lbs 20 lbs 340 yds

## Space Related Cyanate Development Activity

- 1) Amoco 1962 replacement
- 2) 260°F curing adhesive film
- 3) 350°F curing adhesive film
- 4) 350°F curing matrix improved RS-3
- 5) 260°F curing matrix improved RS-12

9

Aracy B-10

AroCy L-10
Character
Chara







### RS-3 Status

Quantity of RS-3 prepreg produced:

Carbon - 2700 lbs

E Glass - 50 lbs

S Glass - 30 lbs

Quartz - 660 lbs

Kevlar - 100 lbs

Total = 3610 lbs

Dow Chemistry: XU71787.02L & XU71787.07L are still experimental. announcement. These materials are now produced in a pilot plant Dow anticipated giving commercial product designation to these materials at SAMPE - 93 in May, but has now postponed this facility.

# Matrix Resins Include:

RS- 1 200°F Curing Modified Epoxy

350°F Service Toughened Polycyanate **RS-3** 

RS-5 350°F Service Polycyanate

RS-6 250°F Curing Modified Epoxy for Aircraft Interior Applications

RS-7 250°F Curing Toughened Epoxy

RS-8 350°F Curing BMI

RS-9 500°F Service polycyanate

RS-11 Latent, Low Moisture Uptake 250°F Curing Epoxy

RS-12 250°F Curing Polycyanate

350°F RTM Toughened Polycyanate RS-14

## Microply Syntactic Films

SF-1 250°F Curing Modified Epoxy Syntactic Film for aircraft interior applications.

F-2 350°F Curing modified Epoxy Syntactic Film for use as structural sandwich core.

SF-3 200°F Curing Modified Epoxy Syntactic film Based on RS-1.

SF-4 BMI Syntactic Film.

SF-5 350°F Curing Modified Polycyanate Based on RS-3.

250°F Curing Toughened Epoxy Syntactic Film. SF-6

SF-7 Phenolic Syntactic Film.

EM-2 Low Smoke, low OSU Expanding Epoxy Syntactic Film

EM-3 Improved 250°F Curing Expanding Epoxy Syntactic Film

500°F Tg Expanding Polycyanate Syntactic Film EM-5



### Cyanate Ester Resin Technology For Aerospace Applications

Prepared By: Bryte Technologies Inc. 1840 Houret Ct. Milpitas, CA (408) 946-6477

> Scott Unger General Manager

> > August 4, 1993



### **Bryte Technologies Corporation Overview**

### **Business Structure**

- Small Business
- Manufacturer of High Technology Prepregs, Adhesives and Resin Systems

### **Technological Market Thrusts for Materials (Niches)**

- Spacecraft
- Aircraft
- Radomes, Antennae and other Electrically Transparent Structures
- Structural Radar Absorbers
- Specialty Commercial. Medical and Other Technologically Advanced Applications

### **Bryte Technologies' Specialties**

- Offering Pertinent Materials and Products to Meet the Focus of Niche Oriented Applications Within Target Market Segments.
- Cyanate Ester Technology in General
  - Bryte Offers More Prepreg, Adhesive and Resin Systems Than any Corporation Worldwide
- Specialty Product Development and Follow on Production of Materials for Specific, Discerning Applications.



### **Bryte Technologies Corporation Overview**

### **Manufacturing Capabilities**

- Hot Melt Prepreg Production
  - a. Unidirectional Tape Production (12" Width)
    - Cured Ply Thicknesses from (0.001 0.015")
  - b. Fabric Prepreg Production (50" Width)
  - c. Non Woven Prepreg Production (50" Width)
  - d. Precision Slit Tow Prepreg Production
  - e. Bulk, Long Fiber Molding Compound Production
    - Epoxy and Cyanate Ester Based
- Hot Melt Cyanate Ester Adhesive Production
  - a. 250°F and 350°F Cure
  - b. Unsupported Film Adhesives (12-36" Width)
    - 0.001 0.015 Thickness (0.006 0.030 PSF Wt)
  - c. Supported Adhesives (36" & 39" Width)
    - 0.035, 0.045, 0.060 PSF Areal Weights
  - d. Paste Adhesives
- Long Fiber Bulk Molding Compound Production
  - a. Cyanate Ester and Epoxy Based
  - b. Carbon, Glass, Quartz and Kevlar® Fibers
- RTM and Filament Winding Resin Systems
  - a. Cyanate Ester and Epoxy Based



### **EX-1515 Prepreg System Background**

• 225 - 250°F Cure Temperature

 Resin System Based Mainly Upon Ciba Geigy's Cyanate Monomer Technology Rather Than Dow Chemical's

· Proprietarily Toughened

### Why EX-1515?

• Optimal Tack, Drape and Flow for Processing Via Autoclave, Oven/Vacuum Bag or Press Cure.

• Unparalleled Conversion Level After the 250°F Cure

- This Assures Microcrack, Radiation and Solvent Resistance.
- Unparalleled Microcracking Resistance
- High Radiation Resistance
- Low Moisture Absorption

Low Outgassing

• Controlled Flow and Toughness Give EX-1515 Self Adhesive Properties and Resultant Co-Curing Ability.

• 250°F Cure Translates Into Lower Residual Stresses in Composite Hardware

 NASA-JPL Testing has Proven a Gain of at Least one Full Order of Magnitude Improvement in Dimensional Stability of 75 Msi, Pitch Graphite Composite Mirror Structures Versus 350°F cured Dow Chemical Based Systems.

• Free Standing Post Cure up to 480°F Increases EX-1515's Glass Transition Temperature without harming its low Residual Stress Advantages Over 350°F Cure Systems.

August 4, 1993



### **Typical Space Applications for EX-1515**

- Dimensionally Stable Space Structures and Optical Benches
- High Technology Reflectors and Solar Arrays for Spacecraft Using Specialty Kevlar® woven goods.
- Low Outgassing. Low Microcracking Composite Structures
- Space Structures Requiring High Radiation Resitance

### **EX-1515 Prepreg Formats**

- Unidirectional Tape Prepregs (.001" .010" Thick)
  - Carbon (Pitch and PAN Based Fibers
  - Kevlar® 49 and 149
  - Fiberglass, Quartz, Spectra®
  - Other (Brytes Will Work With Virtually Any Fiber)

Pitch Car	rbon Fibers	Tensile Stren	igth Tensile Modulus	<b>Strain</b>
Mitsubis	hi KS352U	520 ksi	80 Msi	0.65%
	K1352U	520 ksi	90 Msi	0.60%
	K1392U	520 ksi	110 Msi	0.50%
	K13BU	570 ksi	120 Msi	0.48%
Nippon	XN-50A	530 ksi	75 Msi	0.50%
	XN-70A	530 ksi	105 Msi	0.50%
	XN-80A	530 ksi	114 Msi	0.40%

August 4, 1993



- EX-1515 Prepreg Formats Continued
  - Unidirectional Tape Prepregs (.001" .010" Thick)

Pitch Carbo	on Fibers	Tensile Strength	Tensile Modulus	<u>Strain</u>
Amoco	P75S	300 ksi	75 Msi	0.40%
	P100S	275 ksi	110 Msi	0.25%
	P120S	275 ksi	120 Msi	0.29%

High Mo	d Pan Fibers	Tensile Strength	Tensile Modulus	<u>Strain</u>
Toray	M55J	540 ksi	78 Msi	0.70%
	M60J	570 ksi	85 Msi	0.70%

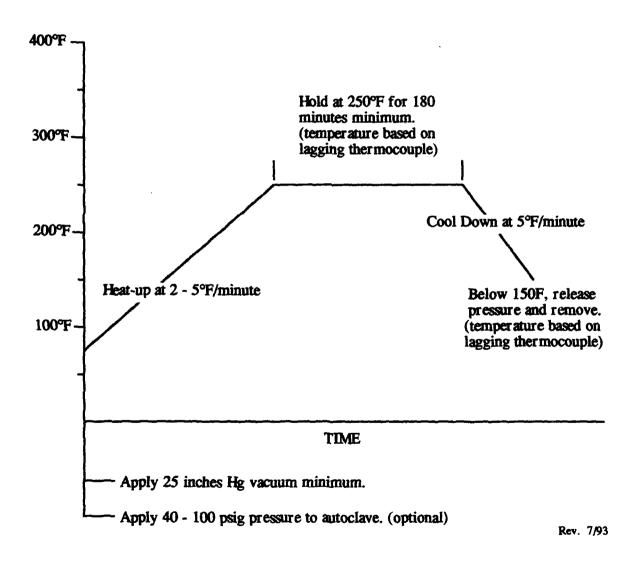
- Fabric Prepregs (To 50" Width)
  - Carbon (Pitch and PAN Based Fibers (All Styles)
  - Kevlar® 49, 149 and Low Moisture Regain (All Styles)
  - Fiberglass, Quartz, Spectra® (All Styles)
  - Other (Bryte Will Work With Virtaully and Woven and Non-Woven Fabric Goods)



Bryte Technologies, Inc. 1840 Houret Ct. Milpitas, CA 95035 (408)946-6477 FAX (408)946-9388

### EX-1515 Cure/Post Cure Cycle

(Cyanate Ester Resin)



Post Cure: (optional)
Heat at 3° to 5°F per minute to 450°F,
Hold at 450°F for 2 hours minimum,
Cool to 160°F and remove.

Exacting Hot Melt Prepregs for the Composites Industry



**EX-1515 Neat Resin Properties** 

• Density 1.16 gm/cc

• CTE 64 ppm/°C above -50°C 34.7 ppm/°C below -50°C

• Moisture Absorption 0.4%

• Outgassing TML 0.179% VCM 0.007%

• Flexural Strength Data Before & After Radiation Exposure

Condition Flexural Strength Modulus
As Cured @250°F 19.2 ksi 0.49 Msi

10<sup>9</sup> Rads Gamma 17.5 ksi 0.48 Msi Radiation Exposure

### **EX-1515 Typical Composite Laminate Properties**

• Fiber Type XN-50A (75 Msi Pitch)

• 0, 30, 60, 90, 120 Sym. Laminate 60% Vf

Mechanical TestTest Value0° Tensile Strength70 ksi0° Tensile Modulus15.3 Msi

0° Compression Strength 23.4 ksi 0° Compression Modulus 11.0 Msi

90° Compression Strength 24.0 ksi 90° Compression Modulus 11.4 Msi



### EX-1515 Resin System

### **PRODUCT TYPE**

225°F – 250°F Cure Toughened Cyanate Ester

**SERVICE TEMPERATURE**230°F Dry Without Post Cure

### TYPICAL APPLICATIONS

- High Dimensional Stability
   Space Structures
- Optical Benches
- Reflectors
- · Radomes and Antennae
- Spectra<sup>®</sup> Composites
- Aircraft Structures
- Low Observables
- Radar Transparent Structures

### **PRODUCT DESCRIPTION**

The EX-1515 Cyanate Ester resin system is very unique among its peers in that it is able to achieve an extremely high level of conversion after a 250°F cure. This level of conversion renders optimal mechanical properties, high radiation resistance, and low moisture absorption/low outgassing while retaining unparalleled toughness, a low, 244°F, stress free temperature and long out time. The resin system excels in its ability to resist microcracking, even when subjected to vicious thermal cycling and high levels of radiation exposure.

EX-1515 also displays low dielectric/low loss values similar to other Cyanate Esters which justifies its use in radome and antenna applications as well. Finally, EX-1515 can be post cured, free standing, to increase its thermal performance for temperature critical structures. Accept no imitations, ask for EX-1515!

### **PHYSICAL PROPERTIES**

Moisture Absorption 0.04% (P75 Laminate Saturation @ 80°F,

and 85% Relative Humidity)

Outgassing TML: 0.179%

VCM: 0.007%

Density 1.16 gm/cc

Resin CTE

Above -50°C 64 ppm/C° Below -50°C 34.7 ppm/C°

### **ELECTRICAL PROPERTIES**

Dielectric Constant 2.8 @10 GHz Loss Tangent 0.004 @10 GHz

### MICROCRACKING DATA

Laminate Type XN-50A Pitch 75 Graphite/EX-1515

Layup Configuration 0/30/60/90/120/150 Symmetrical,

60% Fiber Volume.

Test 1 Conditioned from -112°F to +250°F

for 250 Cycles

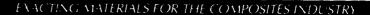
Results 0 Microcracks per Inch

Test 2 Conditioned 25 Cycles in and out of Liquid

Nitrogen, exposed to 1 x 10° Rads of radiation, then Conditioned 50 Cycles in

and out of Liquid Nitrogen.

Results 0 Microcracks per Inch





### EX-1515 Resin System

### **LAMINATE PROPERTIES**

Laminate Type XN-50A Pitch 75 Graphite/EX-1515

Layup Configuration 0/30/60/90/120/150 Symmetrical,

60% Fiber Volume

0° Tensile Strength

Modulus 15.3 msi

0° Compression Strength

23.4 ksi

70 ksi

Modulus

11 msi

90° Compression Strength

24 ksi

Modulus

11.4 msi

Short Beam Shear Strength

6.1 ksi

Laminate Type

XN-50A Pitch 75 Graphite/EX-1515

Layup Configuration

Unidirectional

(Properties Pending)



### Comparitive Data For Bryte Technologies EX-1515 vs. Fiberite 954-3

RESIN	TYPE	CURE TEMP.	
Bryte Technologies EX-1515	Cyanate Ester	250°F	
Fiberite 954-3	Cyanate Ester	350°F	
FIBER TYPE	K1352U	K1352U	
CF Lot No.	19110013	19107001	
Resin	EX-1515	954-3	
Prepreg Made by	Bryte	Fiberite	
Laminate made by	MKC	MKC	
Measured by	MKC	MKC	
LAMINATE PROPERTIES	TEST VALUE EX-1515	TEST VALUE 954-3	IMPROVEMENT WITH EX-1515
0° Tensile (ASTM D3039)			
Strength (ksi)	322	298	24
Modulus (Msi)	54.6	53.9	0.7
Strain (µ in/in)	5700	5300	400
0° Compression (ASTM D3410)			
Strength (ksi)	67	58	9
Modulus (Msi)	45.3	43.1	2.2
0° Flexural (ASTM D790)			
Strength (ksi)	97	89	8
Modulus (Msi)	40.9	40.3	0.6
90° Tensile (ASTM D3039)			
Strength (ksi)	6.7		N/A
Modulus (Msi)	0.8		N/A
Short Beam Shear (ASTM D2344)			
Strength (ksi)	11.4	11.0	0.4



Missiphor Kase American California Office professional Missiphoria 440 Missiphoric CA 4402

### MECHANICAL PROPERTIES OF K1352U/EX1515 UD-LAMINATE

GRADE	K1352U
CF lot No.	19110013
Resin	EX1515
Prepreg made by	Bryte
Laminate made by	MKC
Measured by	MKC
FIBER PROPERTIES	
TENSILE STRENGTH(KSI)	555
TENSILE MODULUS(MSI)	90.9
DENSITY(g/cc)	2.13
YIELD (yd/lb)	1798
SIZING AMOUNT(%)	1.6
LAMINATE PROPERTIES	
0°TENSILE (ASTM D3039)	
STRENGTH(KSI)	322
MODULUS(MSI)	54.6
STRAIN (μ in/in)	5700
OCOMPRESSION(ASTM D3410)	
STRENGTH(KSI)	67
MODULUS (MSI)	45.3
0°FLEXURAL (ASTM D790)	
STRENGTH(KSI)	97
MODULUS (MSI)	40.9
90 TENSILE (ASTM D3039)	
STRENGTH(KSI)	6.7
MODULUS (MSI)	0.8
SHORT BEAM SHEAR(ASTM D2	
STRENGTH(KSI)	11.4

Data normalized to 60% fiber volume except short beam shear strength

Telephone 415-854-5690 Facsimile 415-854-9797



Mitsubishi Kase- America Inc California Office 2180 Sand Hill Road, Suite 440 Mento Park, CA 94025

### MECHANICAL PROPERTIES OF K1352U/954-3 UD-LAMINATE

GRADE	K1352U	K1352U			
CF lot No.	19107001	19104007			
Resin	954-3	954-3			
Prepreg made by	ICI Fiberite	ICI Fiberite			
Laminate made by	MKC	MKC			
Measured by	MKC	MKC			
FIBER PROPERTIES					
TENSILE STRENGTH(KSI)	516	509			
TENSILE MODULUS(MSI)	91.0	89.9			
DENSITY(g/cc)	2.12	2.13			
YIELD (yd/lb)	1844	1865			
SIZING AMOUNT(%)	1.8	1.5			
		i			
LAMINATE PROPERTIES					
0 TENSILE (ASTM D3039)					
STRENGTH(KSI)	298	278			
MODULUS(MSI)	53.9	52.8			
STRAIN (µ in/in)	5300	5000			
0 COMPRESSION(ASTM D3410)					
STRENGTH(KSI)	58	67			
MODULUS (MSI)	43.1	43.2			
0 FLEXURAL (ASTM D790)					
STRENGTH(KSI)	89	92			
MODULUS (MSI)	40.3	39.3			
90 TENSILE (ASTM D3039)					
STRENGTH(KSI)	•	•			
MODULUS (MSI)	•	·			
SHORT BEAM SHEAR(ASTM D2344)					
STRENGTH(KSI) 11.0 10.7					

Data normalized to 60% fiber volume except short beam shear strength

Telephone 415-854-5690

Facsimile 415-854-9797



### DIALEAD TECHNICAL DATA

### FIBER PROPERTIES

		DIALEAD K1352U
Tensile Strength* Tensile Modulus* Ultimate Elongation* Density Carbon Content	KSI MSI % g/cm <sup>3</sup> %	520 90 0.58 2.12 over 99
Filament Diameter Filament Count Yield	μm K yard/lb g/km	10 2 1900 265

Impregnated Strand Test Method



### **Cyanate Ester Materials for Space Applications**

### **EX-1515 Microcracking Data**

Laminate Types

0, 30, 60, 90, 120 Sym

• Fiber Type

XN-50A 75 Msi Graphite

60% Fiber Volume

### <u>Test # 1</u>

• Conditioned from -112°F to +250°F for 250 Cycles

**Results** 

0 Microcracks Per Inch

\* See Photos in Appendix

### <u>Test # 2</u>

• Conditioned in and out of Liquid Nitrogen 25 Cycles

• Exposed to 109 Rads of Gamma Radiation

• Conditioned in and out of Liquid Nitrogen 50 Cycles

Results

0 Microcracks Per Inch



### EX-1515 Microcracking Study



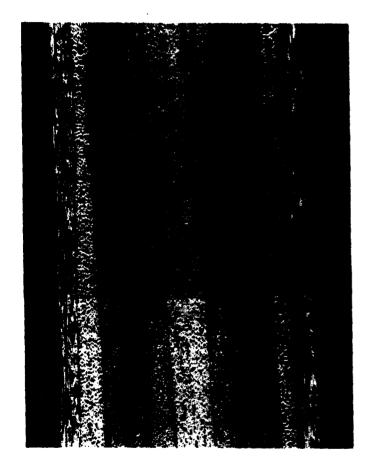
Control Panel (As Cured)
Bryte Technologies'
75 MSI Pitch Graphite/EX-1515
(0, 30, 60, 90, 120, 150) s

50x Magnification

Exacting Hot Melt Prepregs for the Composites Industry



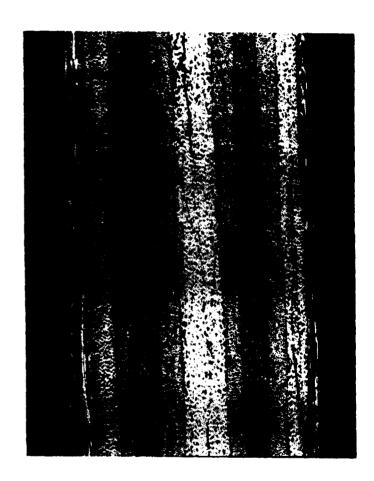
### EX-1515 Microcracking Study



**Bryte Technologies' 75 MSI Pitch Graphite/EX-1515**(0, 30, 60, 90, 120, 150) s

-112°F to + 212°F (25 Cycles)

50x Magnification



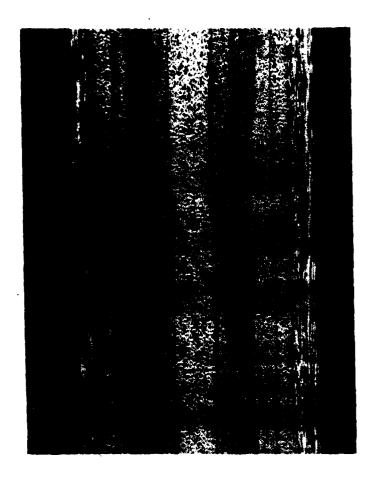
**Bryte Technologies' 75 MSI Pitch Graphite/EX-1515**(0, 30, 60, 90, 120, 150) s

-112°F to +212°F (250 Cycles)

50x Magnification



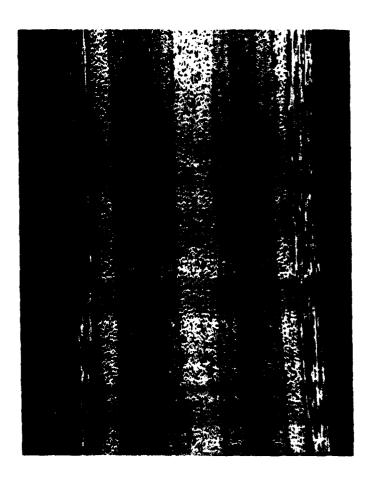
### EX-1515 Microcracking Study



**Bryte Technologies' 75 MSI Pitch Graphite/EX-1515**(0, 30, 60, 90, 120, 150) s

 $-30^{\circ}$ F to + 200°F (25 Cycles)

50x Magnification



**Bryte Technologies' 75 MSI Pitch Graphite/EX-1515**(0, 30, 60, 90, 120, 150) s

 $-30^{\circ}$ F to  $+200^{\circ}$ F (250 Cycles)

50x Magnification



### **Cyanate Ester Materials for Space Applications**

### **Specialty Adhesives for Space and Electrical Applications**

- EX-1516 Toughened Cyanate Ester Film Adhesive (250°F to 350°F Cure)
  - Similar Chemistry to EX-1515
  - Meets NASA Outgassing Specifications
  - Superior Electrical Properties to Epoxy Film Adhesives
- Product Forms
  - Unsupported Films (0.006 0.030 Lbs/Sq Ft Weights)
  - Supported with Polyester, Fiberglass or Quartz (0.035, 0.045, 0.060 lbs.Sq Ft Areal Weight)
- EX-1516 Physical Properties
  - Moisture Absorption 0.6 0.7%
  - Dielectric Constant 2.6 2.7
  - Loss Tangent 0.005 0.006
- EX-1516 Mechanical Properties
  - Fiberglass Non-Woven Supported (0.045 PSF Wt)

Lap Shear 4311 psi on 6061 T-6 Aluminum T Peel 23.6 Lbs/In Width

• Unsupported (0.002" Thickness)

-67°F 77°F 180°F 250°F

Flat Wise 2500 psi 2800 psi 2400 psi 1700 psi Tensile

August 4, 1993



### EX-1516 Film Adhesive

### **PRODUCT TYPE**

250°F – 350°F Cure Toughened Cyanate Ester Film Adhesive

### **SERVICE TEMPERATURE** 250°F Dry

### TYPICAL APPLICATIONS

- High Dimensional Stability
   Space Structures
- Reflectors
- · Radomes and Antennae
- Radar Transparent Structures
- Low Observables
- Aircraft Structures
- Teflon® and Duroid® Circuit Board Bonding

### **PRODUCT DESCRIPTION**

The EX-1516 Cyanate Ester film adhesive has been formulated for use in specific applications where low moisture absorption and/or low dielectric constant/low loss are of utmost importance. Furthermore, the preceding benefits do not come at the expense of adhesion properties. The resin system's strength and toughness when bonding solid, honeycomb or foam core structures is comparable and often greater than high performance epoxy adhesives.

Due to the Cyanate Ester resin system's inherently low shrinkage during cure, bonded structures will retain less inherent stress and will therefore remain dimensionally stable during thermal cycling. This factor is of extreme importance when bonding structures for use in space. Finally, like other Cyanate Ester based products, the EX-1516 film adhesive displays low outgassing and microcracking properties to assure structural integrity even after severe environmental exposure and radiation bombardment.

### **MATERIAL PROPERTIES**

 Moisture Pickup
 0.6 – 0.7%

 Dielectric Constant (10 GHz)
 2.6 – 2.7

 Loss Tangent (10 GHz)
 0.005 – 0.006

### MECHNAICAL PROPERTIES AFTER 250°F CURE (PRELIMINARY DATA)

-67°F

### Non Woven Fiberglass Supported Film

Lap Shear (6061 T-C Aluminum) - 4311 psi T-Peel (6061 T-C Aluminum) - 23.6 lbs/in width (PIW)  Unsupported (.002" thickness)  Lap Shear		•					
width (PIW) – –  Unsupported (.002" thickness)	Lap Shear (6061 T- <b>G</b> Aluminum)	_	4311 psi	-	-		
<del></del>	T-Peel (6061 T-CAluminum)	-		<u>-</u>	- -		
Lap Shear – – – –	Unsupported (.002" thickness)						
	Lap Shear	-	-	-	-		
Flatwise Tension 2500 psi 2800 psi 2400 psi 1700	Flatwise Tension	2500 psi	2800 psi	2400 psi	1700 psi		

77°F

+180°F

250°F

All data given is based on representative samples of the materials in question. Since the method and circumstances under which these materials are processed and tested are key to their performance, and Bryte Technologies Inc. has no assurance of how its customers will use the material, the corporation cannot guarantee these properties.



### **Cyanate Ester Materials for Space Applications**

Specialty Adhesives for Space and Electrical Applications

- EX-1502 Toughened Cyanate Ester Paste Adhesive (250°F to 350°F Cure)
  - Similar Chemistry to EX-1516
  - 2 Part, Thixotropic Paste
  - Superior Electrical and Mechanical Properties to Epoxy Paste Adhesives
  - Will Meet NASA Outgassing Standards

### • EX-1502 Physical Properties

<ul> <li>Moisture Absorption</li> </ul>	0.7%
• Density	1.2 gm/cc
<ul> <li>Dielectric Constant</li> </ul>	2.8
<ul> <li>Loss Tangent</li> </ul>	0.005 - 0.006

### EX-1502 Mechanical Properties

211 1002 Nacchamical 110pcf ties	
• Lap Shear	6000 psi on
-	6061 T-6 Aluminum



### **General Cyanate Ester Technology**

### RTM and Filament Winding Resin Technology

• EX-1510 400 - 450°F Service Cyanate Ester. Ultra Low Viscosity, Excellent Moisture Resistance and Toughness.

• EX-1530 550 - 600°F Structural Service Cyanate Ester. High Char Yield and RTM and Filament Windable at 110°F. Ablative Service to 4000°F+

• EX-1532 550 - 600°F Structural Service Cyanate Ester. Ultra High Char Yield and RTM Capable at 130°F. Ablative Service to 4000°F+

### **Syntactic Foam Technology**

• EX-1541 300°F Cure Syntactic Foam that can be cast, Molded or Machined to Shape.
Lightest Syntactic Foam in Existence

Density
CTE
T - 7.5 x 10-6 in/in/°F (Fully Isotropic)

• Compression Strength 900 - 1000 psi @ 77°F 150°psi, no Crush at 350°F

### **Developmental Capabilities**

• Bryte Specializes in Providing Cost Effective Materials Development to Meet Specific Program Needs



### **General Cyanate Ester Technology**

### **Prepreg Resin Technology**

• BTCy-1	400 - 450°F Service Cyanate Ester for
•	Radomes and High Temperature Structures
<ul> <li>BTCy-1A</li> </ul>	Toughened Version of BTCy-1
• BTCy-2	300°F Service Cyanate Ester for Radomes
•	and Antennae
<ul> <li>BTCy-3A</li> </ul>	Toughened, 250°F Cure Cyanate Ester for
•	Radomes and Spectra® Use
• EX-1505	550°F Service Cyanate Ester With High Char
	Yield. BMI & Polyimide Replacement
• EX-1509	550 - 600°F Service Cyanate Ester with
	With High Char and V-0 Flammability.
	BMI & Polyimide Replacement

### **Adhesive Technology**

•	BTCy-1B	450°F Service Film Adhesive for
	•	Radomes and High Temperature Structures
•	EX-1537	450°F Service, Toughened, Thixotropic
		Paste Adhesive for High Temperature,
		Low Outgassing and Electrically
		Demanding Applications.
	EY_1537_1	450°F Service I ow Viscosity Paste Adhesiy

• EX-1537-1 450°F Service, Low Viscosity Paste Adhesive for High Temperature, Low Outgassing and Electrically Demanding Applications



### Product and Services Selector

**CAPABILITIES** 

• Fabric Prepregs

Up To 50" Width

• Unidirectional Prepregs

Up To 12" Width

Film Adhesives

Unsupported 12" & 24" Width Supported 36" & 39" Width

Non-Woven Prepregs

36 "& 39" Widths

**QUALITY MEASURES** 

• Facility and Processes Qualified to Mil-1-45208

**FIBER CHOICES** 

Fiberglass

E, S2, and D Type

Graphite

Pan and Pitch from 34 to

130 Msi Tensile Modulus

Kevlar<sup>®</sup>Ouartz

29, 49, LMR 49 and 149 Type All Types

• Spectra®

Plasma and Non Plasma Treated

Hybrid and Specialty Fibers

### **EPOXY PREPREGS**

EX-1508

180°F - 250 °F Cure

General purpose Epoxy with 1 Month out time @ 77°F

Applications:

Sporting Goods, Marine, Medical, Low Temperature Curing

Structures, Spectra® Composites.

BT250E-1

250°F Cure

Mil-R-9300 Qualified, Self Adhesive Epoxy for Aerospace and Commercial Use.

3 month out time at room temperature.

Applications:

Secondary Aircraft Structures, Radomes, Sporting Goods,

Medical, Spectra® Composites.

EX-1531

250°F Cure

Quick, 20 minute Cure, Epoxy with Properties Similar to BT250E-1. Specifically Formulated for Commercial Applications to Increase Product Throughput.

Applications:

Sporting Goods, Medical, Commercial Composites.

**BT350E-1** 350°F Cure

Epoxy resin system for high performance applications. BT350E-1 offers excellent

fiber strength translation and good thermal resistance.

Applications:

Aircraft, Missiles, High Temperature Composites

EX-1522 350°F Cure Toughened, Highly Modified Epoxy Hybrid for Critical Hot/Wet Applications. Unparalleled Low Moisture Absorption and Electrical Properties Compared to

Other Epoxies. Self Adhesive to Honeycomb.

Application:

Aircraft, Space Structures, Radomes, Antennae, High Performance

Commercial Applications.

EX-1539

250°F Cure

Flame Retardant Epoxy Prepreg

Applications:

Interiors, Low Smoke/Low Flame Applications.

Revised 3/93

1840 Houret Court ■ Milpitas. California 95035 ■ (408) 946-6477 ■ FAX (408) 946-9388



### RTM/FILAMENT WINDING-EPOXY

EX-1538 350°F Cure High Performance RTM and Filament Winding Resin System. Low Moisture

Absorption, Low Dielectric/Low Loss for Electrical Applications.

Applications: Aircraft, Missiles, Automotive, Radomes, Antennae

### STRUCTURAL RADAR ABSORBING PREPREGS

BT350EX-A 350°F Cure Aerospace Qualified Structural RADAR Absorbing Epoxy Prepreg Material.

Processes and Handles Like Normal Prepreg.

Applications: High Performance Radar Absorbing Structures (RAS)

EX-1525 350°F Cure Flame Retardant Structural RADAR Absorbing Epoxy Prepreg. Meets FAR

Requirements. Processes and Handles Like Normal Prepreg.

Applications: High Performance RADAR Absorbing Structures (RAS)

### **CYANATE ESTER PREPREGS**

BTCy-1 350°F Cure 350°F – 400°F Hot/Wet Service Resin System with excellent structural properties and toughness, low moisture absorption, low outgassing, and very good electrical properties.

Applications:

Aircraft, Missiles, Radomes and Antennae, High Performance

Composite Structures.

BTCy-1A 350°F Cure Toughened version of BTCy-1 which offers improved impact resistance while retaining the benefits of BTCy-1 from a structural and electrical standpoint.

BTCy-1A is self adhesive to honeycomb and foam core.

Applications: Airc

Aircraft, Missiles, Radomes and Antennae, Spacecraft

BTCy-2 350°F Cure Bryte's Best Electrical Performer. BTCy-2 has excellent structural properties and toughness, low moisture absorption, and low outgassing.

Applications:

Ultra low dielectric/low loss radomes and antennae in medium

service temperature environments.

**EX-1505** 350°F Cure

Ultra High Temperature Cyanate Ester with a Tg of 600°F, high char yield, low moisture absorption and very good electrical properties.

Applications:

Aircraft engines, missiles, rocket nozzles, ablatives, heat shields, high temperature radomes, PMR-15 and BMI replacements.

EX-1509 350°F Cure Ultra High Temperature Cyanate Ester with and Tg of 635°F, high char yield, low moisture absorption, V-0 flammability rating and very good electrical properties.

Applications:

Aircraft engines, missiles, rocket nozzles, ablatives, heat shields, high temperature radomes. PMR-15 and BMI replacements.

1840 Houret Court ■ Miloitas, California 95035 ■ (408) 946-6477 ■ FAX (408) 946-9388



BTCy-3 250°F Cure First Generation 250°F Curing Cyanate Ester which displays excellent electrical and structural properties as well as very low moisture absorption.

Applications: High Performance Radomes and Antennae, Spectra®

Composites, Space Structures, Aircraft Hardware, Low Outgassing Composites, and High Performance

Electronic Substrates.

BTCy-3A 250°F Cura Toughened Version of BTCy-3 which offers similar structural benefits with an added dimension of toughness and improved self adhesive properties to honeycomb. BTCy-3A also offers slightly improved electrical performance compared to BTCy-3.

Applications: High Performance Radomes and Antennae, Spectra®

Composites, Space Structures, Aircraft Hardware, Low Outgassing Composites, and High Performance

Electronic Substrates.

EX-1515 225°F-250°F Developed specifically for NASA, this 2nd generation, low temperature curing, toughened Cyanate Ester matrix has ultra high conversion, extremely low moisture absorption, low outgassing, high radiation resistance and is fully self adhesive. Its low temperature cure coupled with its unparalleled resistance to microcracking assure that EX-1515 will render hardware that displays the utmost in dimensional stability. Finally, EX-1515 can be post cured free standing up to 450°F for increased thermal resistance.

Applications: Spacecraft and Stable Structures, Reflectors, Aircraft, Structures

at Cryogenic Temperatures, and Radomes.

### **CYANATE ESTER ADHESIVES**

**EX-1516** 250°F Toughened Cyanate Ester Film Adhesive with outstanding electrical performance, and low moisture absorption, and low outgassing without affecting structural performance.

Applications:

Radomes, Antennae and other microwave applications, Space and Aircraft Structures, bonding for cryogenic structures.

**BTCy-1A** 350°F

Toughened Cyanate Ester Film Adhesive with very good electrical performance, low moisture absorption, low outgassing and very good thermal resistance.

Applications:

Radomes, Antennae and other Microwave Applications, Space and Aircraft Structures, High Service Temperature Structures.

**EX-1502-1** 250°F-350°F

Toughened Cyanate Ester Paste Adhesive that offer similar properties to EX-1516 coupled with 6000 psi lap shear on aluminum.

Applications:

Radomes, Antennae and other Microwave Applications, Space and Aircraft Structures, Bonding for Cryogenic Structures.



### EXACTING MATERIALS FOR THE COMPOSITES INDUSTRY

**EX-1537** 350°F Cure

### CYANATE ESTER ADHESIVES CON'T.

Toughened Cyanate Ester paste adhesive with high thermal resistance and excellent structural properties.

Applications: Radomes, Antennae and other Microwave Applications, Space

and Aircraft Structures, High Service Temperature Structures.

### CYANATE ESTER FILAMENT WINDING AND RTM RESINS

**EX-1510** 350° Cure

Low viscosity RTM and filament winding matrix. Room temperature processable for RTM and filament winding. EX-1510 offers low moisture absorption, high Tg, good electrical properties and very long pot life.

Applications: Aircraft and Space Structures, Radomes, Antennae.

350°F hot/wet performance.

**EX-1530** 350°F Cure

2 part, high service temperature matrix which is processable at 110° – 120°F. EX-1530 offers high char yield, low moisture absorption, excellent ablative characteristics, 8 hour pot life and good electrical properties.

Applications:

Aircraft, Aircraft Engines, Missiles and Rockets, Ablatives, and

High Temperature Radomes.

**EX-1532** 350°F Cure

2 part, high service temperature matrix which is processable at 130 – 140°F. EX-1532 offers high char yield, low moisture absorption, excellent ablative characteristics, 4 hour pot life and good electrical properties. It differs from EX-1532 in that it is slightly higher in viscosity and has a higher Tg and char yield.

Applications:

Aircraft, Aircraft Engines, Missiles and Rockets, Ablatives,

and High Temperature Radomes.

### PHENOLIC, POLYIMIDE & BISMALEIMIDE

Bryte Technologies can also provide products based on these matrices.

### **BULK MOLDING COMPOUNDS**

Bryte Technologies will soon have a line of Long Fiber, Bulk Molding compounds that are optimum for compression molding of intricate composite hardware for Commercial, Sporting Goods and Aerospace Composites.



### **Conclusion**

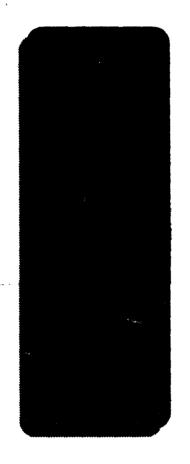
### **Technology**

- Bryte Offers the Widest Array of Cyanate Ester Prepregs, Adhesives and Resin Systems to Meet Many Aerospace Needs.
- EX-1515 Resin Along With Associated EX-1516 and EX-1502 Adhesives Offer a Systematic Approach to the Structural and Physical needs of Spacecraft
- EX-1515 is Unparalleled With Respect to Dimensional Stability, Low Microcracking, Radiation Resistance, Polymer Conversion after 250°F Cure Cycle and Co-Curing Ability

### **Service**

• Bryte Technologies Quick Lead Times of 2-4 weeks, and Cost Effectiveness Provide the Aerospace Community With a Reliable High Technology Materials Source to Meet The Stringent Cost Structure and Reactive Nature of Today's Programs.

### APPENDIX E COMPOSITE PARTS FABRICATOR PERSPECTIVES



JUNE 16, 1993

SPARTA, INC. 9455 TOWNE CENTRE DRIVE SAN DIEGO, CA 92121-1964 TEL: (619) 455-1650 FAX: (619) 455-1698



### SPARTA HAS FOCUS\$ED CYANATE DEVELOPMENT ON INTERCEPTOR STRUCTURES

e-salpadin

Sponsored by John Dignam at Army Research Lab-Materials Directorate

Agent: USASSDC / Key Technologies

Funding by: BMDO/DTI

Why Cyanate Ester Resins for Interceptor Structures?

- Low Moisture Absorption Minimize Outgassing During Flyout and Exo
- Ease of Processing Compatible with Matched Metal Net Mold Process
- Prepreg Available in High Modulus (100 Msi) Pitch Fibers Interceptors are Stiffness Office
- Interceptors such as TMAAD Higher Operating Temperatures (with post cure) — Important for Endo

### Project Scope:

- Evaluate Graphite Fiber / Cyanate Prepreg for Use by Matched Metal Matalian Metal Metal
- Eabricate High Modulus Graphite / Cyanate Ester Structures for Full-Maile Testing



## YLA RS-3 POLYCYANATE MOISTURE ABSORPTION TESTING

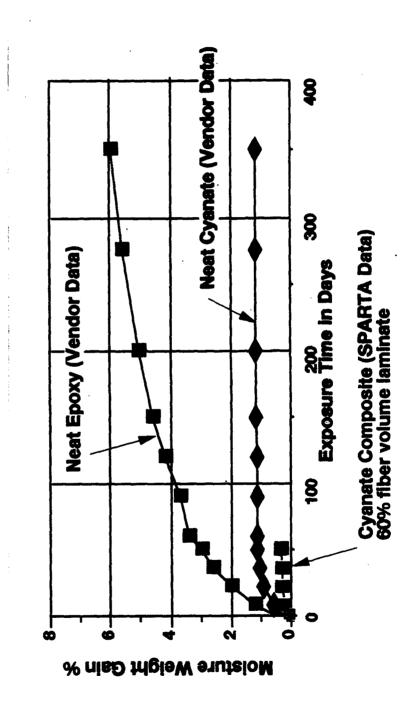
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TASK

Fabricated Panels and Conducted Moisture Absorption Tests

NOIST ENCO

Vendor Reported Moisture Absorption Validated





## ICI/FIBERITE 954 CYANATE ESTER RESIN EVALUATION

SPAKIA

Fabricated 6" Long Cylinders

- FT700 Graphite Tape - FT700 Graphite Cloth
- Conclusion
- SEF-3 Resin Consistency Like Water During
  - Runs out of Mold Causing Resin Starved Aress

Fabricated 6" Long Cylinders

- FT700 Graphite Tape

- FT700 Graphite Cloth

Conclusion

954-2A Resh has Higher Viscoetty This and Produced High Quality Parts



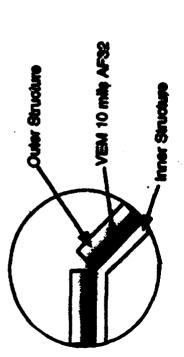
### GRAPHITE FIBER/CYANATE ESTER GBI STRUCTURES **WITH PASSIVE DAMPING**

4/LED/65-9

Manufacture Graphite Fiber/Cyanate Ester Structures With Mold-in-place Constrained Layer Visco-Elastic Material (VEM)

HOYORLinks

Achieve Enhanced Damping with A "Sandwich" of Graphite/Cyanate Ester Facesheets and a VEM Core



Outer Layer of Sandwich is Silt, Which Allows Differential Shear of Cylinder (Thin Walled Cylinder Normally Exhibit Only Membrane Tension/Compression Strain)

1/1

3M AF-32 has 350°F Cure Cycle That Matches Cyanate Ester Cure Cycle



## STATUS OF GBI STRUCTURES WITH PASSIVE DAMPING

DAMPED GEI STRUCTURE Generic GBI structure being fabricated in June 1983 Model Test in July 1993 · Fabricated Undamped & Damped Cylinders Seed New (21 Ba) for 190 mai floor batterned Companies MANUFACTURING DEVELOPMENT -- CYLHIDER SELLING DEVELOPMENT --1 MANUFACTURING DEVELOPMENT --PANEL

Undamped, 3% damping, 200 Hz
 Damped, 5% damping, 200 Hz

Showed Co-curing of silts

 Undamped & Damped Panels Fabricated

Metallography showed
- Uniform 10 mil VEM
- Low void content

Model Survey



## SUMMARY OF SPARTA'S CYANATE RESIN DEVELOPMENT

enflirates-s

# SPARTA'S WORK WITH CYANATE RESINS HAS SHOWN:

- LOW MOISTURE ABSORPTION (APPROX 1% FOR NEAT RESIN)
- GOOD PROCESSING BEHAVIOR IN A MATCHED METAL NET MOLD FOR IC/FIBERITE CYANATE ESTER 954-2A
- GOOD QUALITY PREPREG AND LAMINATES WITH HIGH MODULUS GRAPHITE PITCH FIBERS (100 MSI MODULUS)
- MOLD IN PLACE VEM MATERIAL SUCCESSFULLY DEMONSTRATED
- CANDIDATE BMDO/ARMY APPLICATIONS FOR CYANATE ESTER
- Ground Based Exo Atmospheric Interceptor KKVs (Low Outgassing)
- THAAD, Corps SAM, and ERINT (Higher Temperature Requirement)
- Ground Based Radar Antenna Transmit Unit (Low Dimensional Distortion)



## PHILLIPS LABORATORY



# GRAPHITE REINFORCED POLYCYANATE OVERVIEW

CLEMENTINE

JAMES L. KOURY

IDA WORKSHOP

16 JUNE 1993



## PHILLIPS LABORATORY



### INTRODUCTION

BACKGROUND

BTCy--1 RESIN SYSTEM

BTCy--1 CURE / POST CURE CYCLE

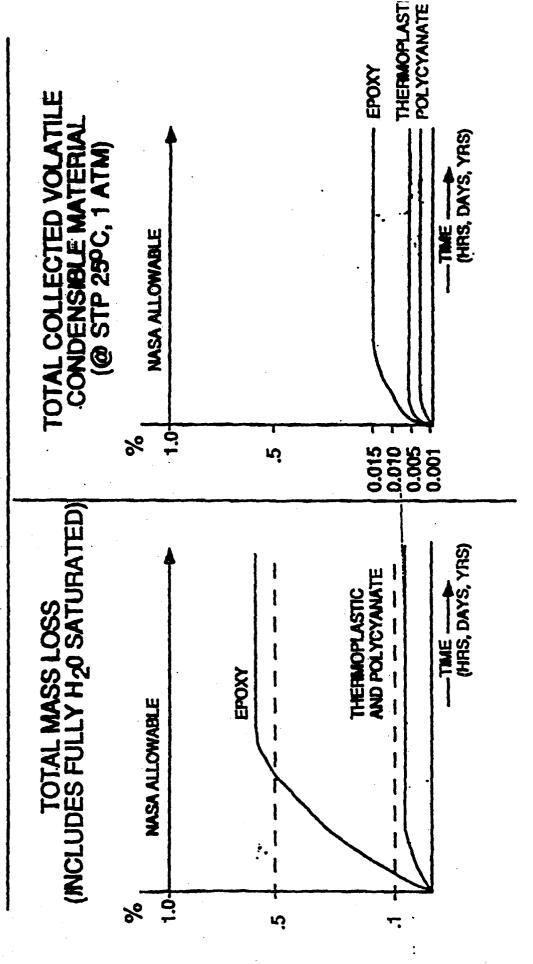
MATERIAL CHARACTERIZATION

COMPONENTS

CONCLUSIONS / RECOMMENDATIONS



### OUTGASSING OF RESIN COMPOSITES FOR SPACECRAFT APPLICATION



SYSTEM	CURE TEMP.	Tg	Н 20	APPLICATIONS
DGW XU - 71787	350 F	290 C	0.6-1.2	RADOMES, ANT SPACE STR.
FIBERITE ICI 934 SERIES	350 F	260 C	0.2-0.6	SAME AS ABOVE LONG LEAD TIME
BASF X6555 5575	350 F	250 C	1.0-1.3	SAME AS ABOVE Long Lead Time
AMOCO ERL 1939	350 F	240 C	0.9-1.6	SAME AS ABOVE
YLA RS-3	350 F	264 C	1.4	SAME AS ABOVE
B T C y 1	350 F	260 C	0.6 - 1.0 QUICI OWN	- 1.0 SAME AS ABOVE QUICK RESPONSE DOES OWN PREPREG



### PHILLIPS LABORATORY





### NEAT RESIN PROPERTIES

 $\mathbf{T}_{\mathbf{c}}$ 

H O ABSORPTION DIELECTRIC

LOSS TANGENT

G1C

SERVICE TEMP

518° F

1% @ 100 C SAT

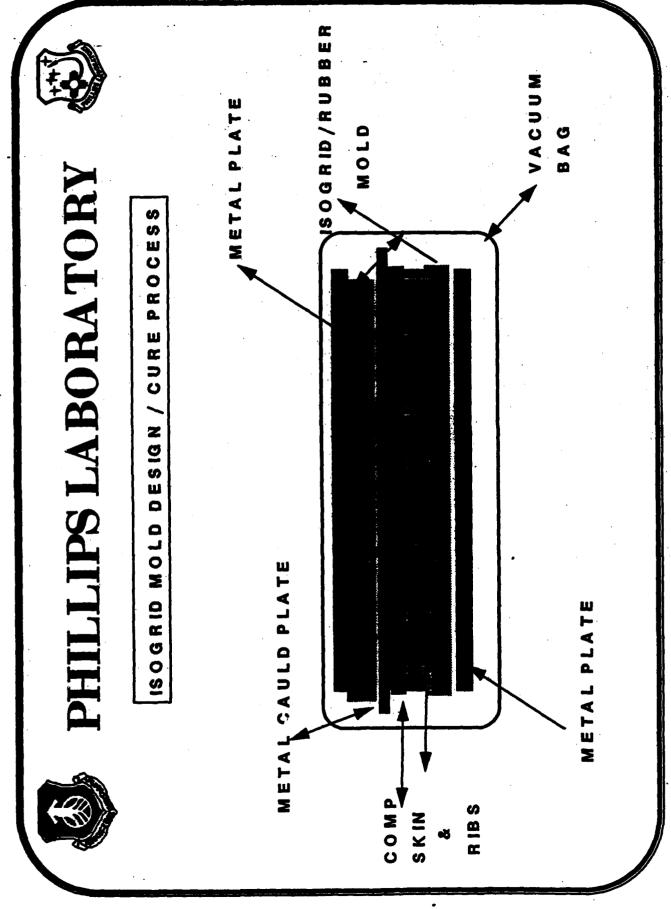
2.7-2.8 (FLAT TO 18 GHZ)

0.003

1.2 IN- LBS / IN

350'F HOT/WET

500°F + SHORT TERM



### PHILLIPS LABORATORY 80 PSIG PRESSURE APPLY VACUUM TIME 300°F 400\*F 200°F

# MATERIAL CHARACTERIZATION

# TOXYTEX INROWIDIECT AND ANTO LOSTINOTRANDING TO PAUNICIL

F.O. (0°)				F.O. (0°/90°)				E.O. (0°/90°)
S SRM 1-88	MODULUS	MSI (STD)	18.04 (0.25)	<b>LUSSRM 1-88</b>	MODULUS	MSI (STD)	11.14 (0.17)	<b>VGTHSRM 1-88</b>
O' COMPRESSION MODULUS SRM 1-88	WIDTH THICKNESS	N	0.047	0°/ 90° COMPRESSION MODULUS SRM 1-88 F.O. (0°/90°)	THICKNESS	Z	0.061	0°/ 90° COMPRESSION STRENGTHSRM 1-88 F.O. (0°/90°)
O COMPRE	WIDTH T	Z	0.502	0% 90° COMP	WIDTH	Z	0.500	0°, 90° COMP

COMP STRENGTH

MAX LOAD

WIDTH THICKNESS

LBS

3,057

0.0622

0.502

KSI (STD) 98.1 (5.0)

			· · · · · · · · · · · · · · · · · · ·
	POISSON RATIO 0.281 (STD) (0.02)	9	STRAIN  5,643 (STD) (303) (45°, 135°)2  R POISSON LUS RATIO ( 0 0.881 0) (STD) (STD) (STD) (STD) (O0057)
8(0)8	TENSILE ULTIMATE TRENGTH STRAIN KSI U IN/IN 349 12,151 (STD) (STD) (15.2) (765)	F.O. (90°)16	ENT OLUS SI SI 99 99 99 95) 95) MODUI MSI 0.55( (0.016
ASTM - D 3039 F.O. (0°)8	TENSILE TENSILE U MODULUS STRENGTH MSI KSI 22.87 349 (STD) (STD) (STD) (STD)		D3 VISI VISI VISI VISI VISI VISI VISI VIS
STM-D	MAX LOAD TENSILE LBS MODULUS 8,215 MSI 22.87 (STD) (0.53)	ASTM - D 3039	
	ייי גע כו	RTIES	S MAX LOAD TEN LBS STRI LBS STRI (6 (6 (7 (7 (1)184 KSI 1,184 KSI 1,184 KSI 1,184 KSI (6.9)
E PROPE	THICKNESS IN 0.0468	E PROPE	THICKNESS IN 0.0917 ICKNESS M IN 0.0489
O° TENSILE PROPERTIES	WIDTH TIN 0.502	90° TENSILE PROPERTIES	## WIDTH THICKNESS MAX LOAD TO INOUS 0.0917 502 ST 502  ### THICKNESS MAX LOAD TENSION / INPLANE SHEAR IN LBS STRENG IN IN LBS STRENG INOUS 0.0489 1,184 KSI (STD (0.9))
0		90,	E-16 +1



## PHILLIPS LABORATORY



## CONCLUSION / RECOMMENDATIONS

**EXCELLENT PROPERTIES** 

PANELS MET ALL REQUIREMENTS EXCEPT FLATNESS

TOW PREPREG VARIABILITY DIDN'T AFFECT QUALITY OF PART

RI RECAUSED WARPAGE

FLEMS USED TO DETERMINE OPTIMUM CURE CYCLE

TEMPERATURE DIDN'T VARY 10 -- 15 F FROM BOTTOM OF RIB TO TOP OF SKIN

## Gr/Polycyanate Composites

## - Processing Perspective

### Martin Marietta Astronautirs, Denver, CO Suraj Rawal

#### Outline

Current Experience

Processing - Related Issues

- Technical

- Technological

Potential R&D Directions

#### Purpose

To Establish the Requisite Heritage and Users Confidence to Insert Gr/Polycyanate Composites into SDIO/DOD/NASA Systems

206.3

#### MINDTIN MINDIETTIN

#### **Gr/Polycyanate**

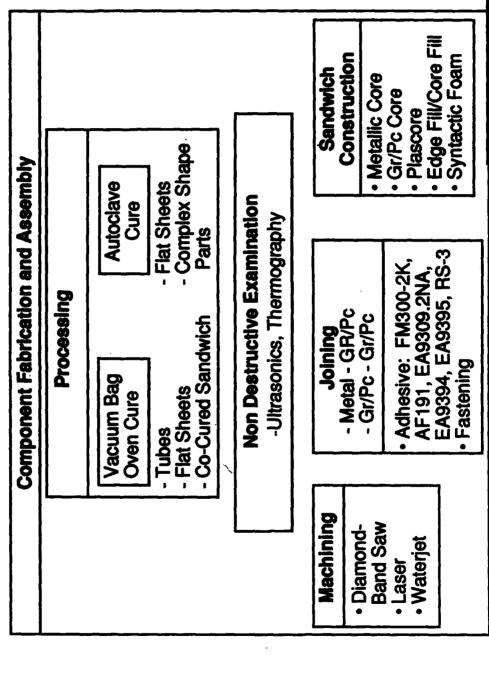
### **CURRENT EXPERIENCE**

- Gr/Pc Composites Offer increased System Robustness and Reliability
- Fabrication of Components and Demonstration Structures for Space **Applications**
- Flat Panels
- **Tubes**
- Honeycomb Sandwich Panels
- Demonstration Structures/Thermal Management Components
- SAWAFE Panels

28

# Composite Parts Fabrication: Gr/Polycyanate

Martin Marietta Experience



## SAWAFE Integrated Sensor Panel

Spacecraft Attack Warning and Assessment Flight Experiment

Integrate RF, Laser, and X-Ray Detectors in Structural Panel

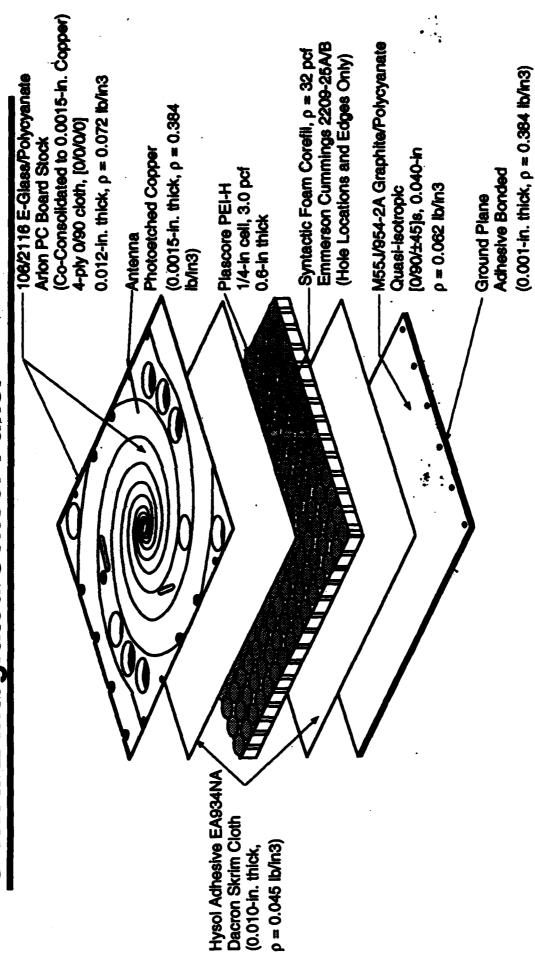
Thermo Detector Cav - RF Copper Antenna Combination Detecto Cavity Pyro Detector-Cavities, 5 PLCS Space Test Experiment Platform

(STEP-3)

FURAR FOR A + 100 SERVER SEALER

R&T, Mechanical

## **SAWAFE Integrated Sensor Panel**



NY ARBY X 8186 BY ARRIVER BRANK

R&T, Mechanical

P120/RS-3 [0/90]s-Aluminum Honeycomb Baseplate/Radiator

## Gr/Polycyanate Composites

Prepregs/Supplier

#### **Fiberite**

- M55J/954-2A
   M60J/954-2A
  - UHMS/954-2A

**Bryte Technology** 

T300/BTCy-1

- XN50/RS-3 XN70/RS-3
  - P100/RS-3
    - P120/RS-3 112E/RS-3
- T300/RS-12 T300/RS-3

#### **AMOCO**

• P100/1999

Generated Material Property Data Base (of Select Few Composites)

## Processing: Gr/Polycyanate Composites

⇒ Processing-Related Issues

None: Tooling and Process Conditions Similar to Epoxy Matrix Composites

Vendor Recommended Autoclave Cure Process Yielded Well Consolidated Parts

⇒• Concerns
Prepreg

Inconsistency in Resin Content (%)⇒

Very Low Tack (Sometime)



Adhesives

Non-Uniformity in Polycyanate Film Adhesives

MAINTIN MAININETTE

## **Gr/Polycyanate Composites**

- Technological Issues/Concerns
- ⇒ Material Supply
- Recent Scare of Precursor Matrix Shortage
- Need to Know Matrix- Producers (Dow Chemical & Ciba Geigy) View
- ⇒• Any Perceived or Real Toxicity Concerns During Matrix and Prepreg **Processing**
- Shouldn't Become the Beryllium of Thermoset Composites
- ⇒• Have the Matrix Formulations Reached a Steady State? (e.g., 954-2—• 954-2A---!)

## **Gr/Polycyanate Composites**

Potential R&D Directions

### Fundamental and Applied R&D

### Structure—Properties Relationship

- Interface Structure Properties
- Additives/Modifiers- Toughening Mechanisms
- Matrix Outgassing/Microcracking Resistance

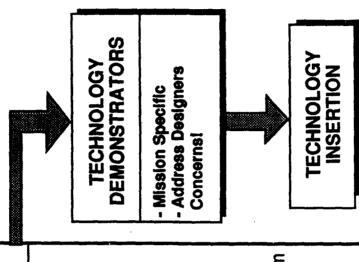
#### Design Data Base

- Material Property Data
- Fatigue Life/ Fracture Toughness
  - Damage Tolerance

- Processing
  Additives-Rheology-Tack-Processing Window
  Consistent v/o Control of No Bleed Resin Sytem
  - Design of Experiment for Low Cost Processing
    - Intelligent Processing

#### Joining and Assembly

- Cyanate-Ester Adhesives
- Adherence of Environmental Coatings



#### Gary Krumweide Composite Optics

Workshop on Graphite-Reinforced Polycyanate Composites

Institute for Defense Analyses Alexandria, VA

June 16, 1993



#### CYANATE SYSTEMS

### **EXPERIENCE 1989 - PRESENT**

MATERIAL TYPE (Cyanate Ester)	P75S/954-3 P75S/954-3	P75S/ERL 1999	P75S/ERL1999	K149/EX1515	K149/EX1515 XN50 Fabric/RS3 P75S/ERL1999	K149/EX1515
MATERIAL M.	500 150	180	30			•
OIX	~ ო	ო	<del></del>		2-2	1
HARDWARE TYPE	1. STRUCTURES Telescope & Bezeis	Telescope	2. SOLAR PANEL Clementine	R&D (Coupons)	3. REFLECTORS (Assembly) R&D (H/C) R&D (H/C) CRAD (H/C)	R&D (Coupons)



#### CYANATE SYSTEMS

### **EXPERIENCE 1989 - PRESENT**

MATERIAL TYPE (Cyanate Ester)	P75S/ERL 1999 XN50/954-3	XN50/RS3	T300 Fabric/RS3	XN70/RS3 & XN50/RS3
MATERIAL USAGE (Ibs)	1 1	ı		•
QIX	- 2	7	IDES 10	10
HARDWARE TYPE	4. MIRRORS SAO (Submil) H/C JPL (1-micron)	5. PHASED ARRAY R&D	6. FEED HORNS/WAVEGUIDES R&D / CRAD	7. MUX CAVITIES CRAD



#### **GENERAL:**

## MATERIAL PROCESSES SIMILAR TO EPOXIES OR MODIFIED EPOXIES

- LAY-UP CHARACTERISTICS AS GOOD OR BETTER THAN EPOXIES
- FLOW DURING CURE HIGHER THAN EPOXIES (Special Attention to Bagging)
- TACK SIMILAR TO EPOXIES
- CURES LIKE EPOXIES (HEAT-UP PROFILE)



#### MECHANICAL PROPERTIES:

- **QUASI-ISOTROPIC LAMINATE PROPERTIES NEARLY EQUIVALENT TO BASELINE EPOXIES** (i.e., E<sub>T</sub>, F<sub>TU</sub>, F<sub>CU</sub>, etc.)
- HIGH RESISTANCE TO MICROCRACKING
- MICRO-YIELD VALUES SIMILAR TO EPOXIES
- COMPRESSION AND SHEAR MODULUS UNAFFECTED BY IRRADIATION (2M RAD AND **200M RAD)**
- INTERLAMINAR PROPERTIES SLIGHTLY LOWER THAN MODIFIED EPOXIES (i.e. ERL1962)



#### PHYSICAL PROPERTIES:

- HIGHER Tg (350°F) Tg BASED ON POST CURE TEMPERATURE
- CTE (a) MORE NEGATIVE THAN SAME EPOXY LAMINATE
- P75S/EPOXY ISOTROPIC 0.00 ± 0.10 ppm/°F
- P75S/CYANATE ISOTROPIC -0.10 ± 0.10 ppm/°F
- CTE LINEAR OVER A GREATER TEMPERATURE RANGE
- CME VALUES GENERALLY THE SAME AS EPOXIES, BUT ASSOCIATED STRAIN SIGNIFICANTLY LESS (1/3)
- MOISTURE ABSORBED SIGNIFICANTLY LESS (1/3) THAN SAME EPOXY LAMINATES
- RATE MOISTURE ABSORBED IS 5-10 TIMES FASTER THAN SAME EPOXY LAMINATES



## CYANATE VS. EPOXY

PIC	CYANATE	14.5	10.0	12.25	4.25	0.33	0.061	-0.1 TO -0.3
P75S / RESIN - ISOTROPIC	EPOXY	15.1	13.1	14.1	5.2	0.32	0.062	-0.08 TO -0.15
P75S /	PROPERTY	ET (MSI)	Ec (MSI)	EAVG(MSI)	G (MSI)	NC	DENSITY (PCI)	ALPHA (PPM/F)



## CYANATE VS. EPOXY

### P75S / RESIN - ISOTROPIC

CYANATE	45.0	25 - 27	15 - 20	2.0
EPOXY	47.3	28 - 31	19 - 23	2.7
PROPERTY	FTU (KSI)	F <sub>CU</sub> (KSI)	FSU (KSI)	FWT (KSI)

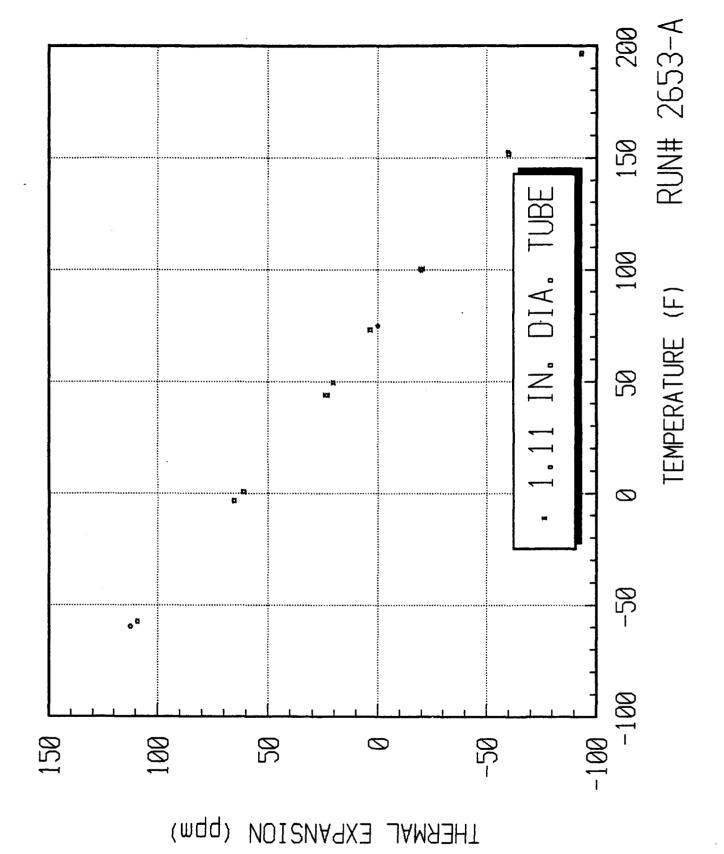
WIP TESTING SUMMARY P75S/ERL1999 (0,45,90,135)<sub>s</sub>

LAB NO. 4265	E (MSI) 14.4	F <sub>TU</sub> (KS!)	SBS (KSI) 4.5	RC [%] 28.6	CTE (PPM/°F) 03	t (IN)
4265 4295	14.6 13.9	38.2	4.5 5.7	27.5	02	.039
4295	14.6	42.4	4.9	27.3	.01	.042
4322 4322	14.1 4.2	45.5 52.6	5.4 4.	28.2	08 05	.041
4353	14.1	•	5.9	32.7	08	.040
AVERAGE	14.3	43.6	5.1	28.8	06	.041

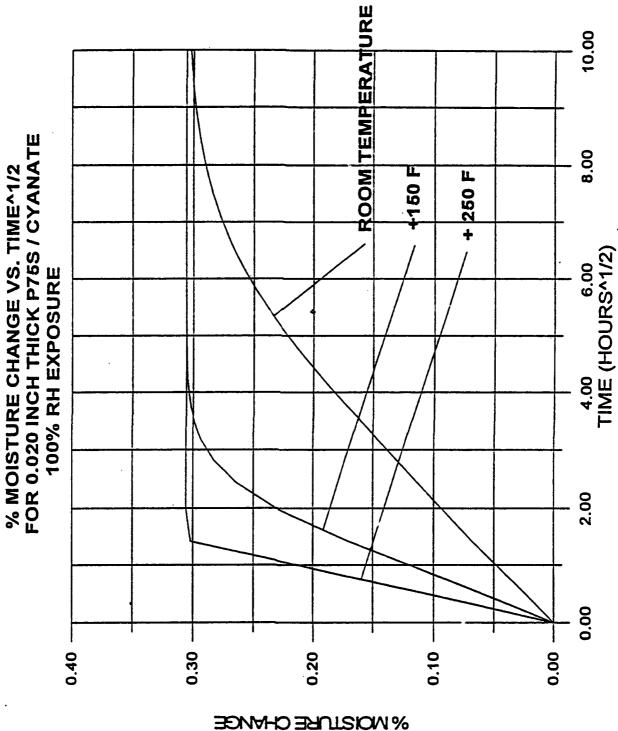


## -- MECHANICAL TESTS - IRRADIATED SPECIMENS

•	LOT 03052	LOT 03053
COMPRESSION MODULUS (MSI)		
2MRAD	8.51	8.85
200MRAD	9.00	8.64
IOSIPESCU MODULUS (MSI)		
2MRAD	4.18	4.64

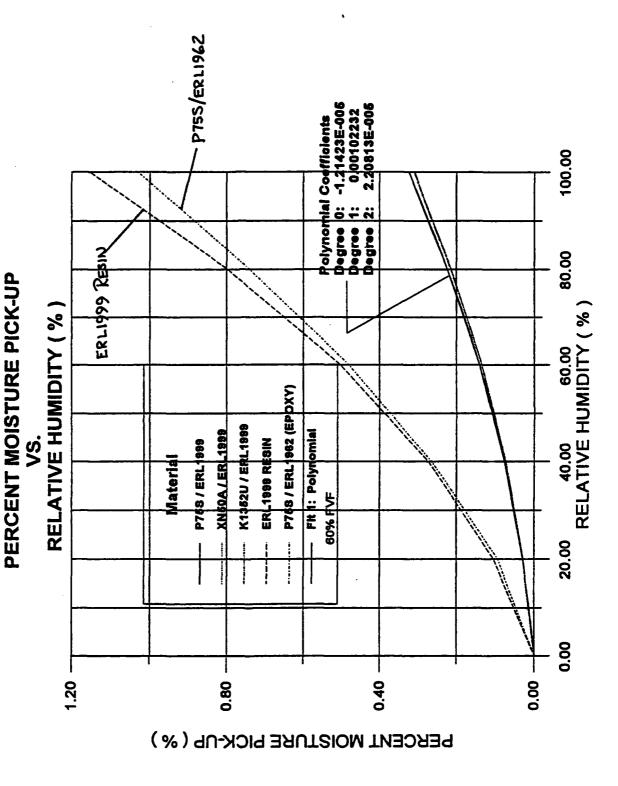






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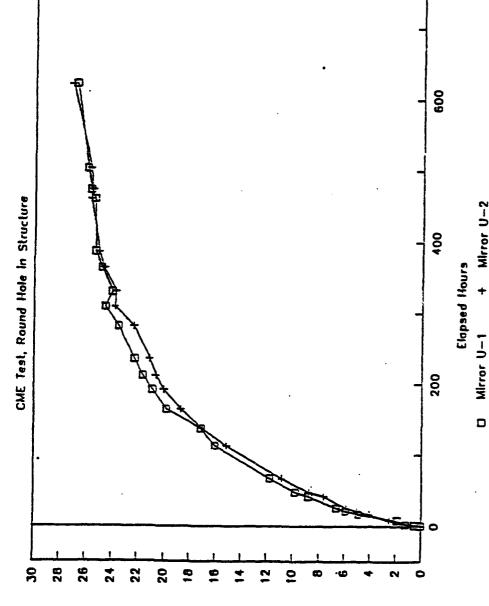






#### -- CME TESTING --

#### PROTOTYPE STRUCTURE





## **BONDLINE ALLOWABLES**

## **▶ PSEUDOISOTROPIC P75S / RESIN**

BUTT TENSION (LBS./IN.)	EPOXY BS./IN.)	CYANATE 154
(0.06")	302	177
(0.08")	366	208
<b>BUTT SHEAR (LBS./IN.)</b>	S./IN.)	
(0.04")	297	268
(0.06")	381	372
(0.08")	449	408
LAP SHEAR (PSI)		
(GR-GR)	1458	1018
(GR-TI)	984	1103



#### OTHER

- GOOD SOLVENT RESISTANT IN CURED STATE
- PRESENCE OF MOISTURE PRIOR TO AND DURING CURE AFFECTS CO-CURE BOND STRENGTH (i.e. KAPTON)
- **LOWER RESIN MODULUS**
- NEAT RESIN STRAIN TO FAILURE HIGHER
- **NEAT RESIN CTE HIGHER**
- LOW DIELECTRIC CONSTANT
- **MORE UV RESISTANT**
- LONG-TERM THERMAL STABILITY (BETTER THAN EPOXIES)
- **HIGHER COST**
- LIMITED USAGE ON FLIGHT HARDWARE



#### CONCERNS

CYANATE RESINS HAVE SHOWN AN EFFECT TO POST **CURING EFFECTS** 

♦ NEEDS TO BE VERIFIED

NEED TO GENERATE MORE CONSISTANT MOISTURE DATA FOR CYANATES

◆ CME (PPM/%M)

♦ %M VS. RH

♦ DIFFUSIVITY VS. TEMPERATURE



## CLEMENTINE SOLAR PANEL SUBSTRATES MATERIAL/PARTS LIST

	ITEM	MATERIAL DESCRIPTION	VENDOR	COMMENT
	SKINS & DOUBLERS	P75S/ERL1999 (2K) FAW 80.2G/M²(R/C36.6)	АМОСО	PROCURED BY PHILLIPS LAB (4-ply Skins & Doublers)
	HONEYCOMB CORE	5056AL, 1/8 CELL, .0007 3.1 LB/Ft³, PERFORATED	PLASCORE	1
٠	KAPTON FILM	200FPC, .002 ETCHED BOTH SIDES	DUPONT	ONE SURFACE OF PANEL
	ADHESIVE	FM 300-U,.03 LB/Ft <sup>2</sup> MMM-A-132A, TYPE2,GRP4	AMERICAN CYANAMID	SKIN/CORE BOND
	INSERTS	TITANIUM, 6AL-4V	ŧ	COI FABRICATED
	POTTING COMPOUND	CORFIL 615, DTA CURING AGENT	AMERICAN CYANAMID	•
	ADHESIVE	EA 9394	HYSOL	INSERT INSTL
	KAPTON TAPE	T-188-2-1	FRALOCK	SCOTCH HITEMP ACRYLIC (PS)



### CLEMENTINE SOLAR SUBSTRATE CONSTRUCTION FEATURES

- KAPTON FILM CO-CURED TO GR/CE SKINS
- H/C CORE BOND UTILIZED COI RETICULATION PROCESS
- CORE FILL COMPLETED AFTER SKIN/CORE BONDING
- **EDGE CLOSURES WITH KAPTON TAPE**
- INSERTS BONDED AFTER PANEL COMPLETED
- ACCELERATED CURES OF COREFILL AND EA9394 ADHESIVE
- DOUBLERS SECONDARY BONDED TO OUTER SURFACE



#### **CLEMENTINE SOLAR SUBSTRATE** REQUIREMENT/VERIFICATION

FEATURE	REQUIREMENT	ACTUAL MEASUREMENT
FLATNESS	A) .010 IN 12 X 12 AREA B) .030 OVERALL (MAX)	A) .001004 B) .005021
PARALLELISM	A) .010 IN 12 X 12 AREA B) .030 OVERALL (MAX)	A) .001004 B) .003016
WEIGHT INBOARD OUTBOARD	A) 2.8 LBS. B) 3.7 LBS.	A) 2.56 AVG. B) 3.18 AVG.
INSERT PULLOUT STRENGTH	<ul><li>A) 225 LBS.</li><li>AT ROOM TEMPERATURE</li><li>AFTER THEMAL CYCLING**</li></ul>	A) COI (357 - 397 LBS B) PHILLIPS LAB (350-390) LBS.

<sup>\*</sup> PANELS THERMAL SHOCKED USING LN<sub>2</sub> \*\* PANELS SURVIVED THERMAL CYCLING TEST AT NRL (-200°C TO + 94°C)





R. A. Lewis 16 June 93

# POLYCYANATE MATRIX PREPREG FABRICATION EXPERIENCE

### RESEARCH BEGAN 1988

MITSUBISHI	K - SERIES FIBERS	139 (100 MSI)
DUPONT	E - SERIES FIBERS	75/130 MSI
AMOCO	P - SERIES FIBERS	75/100/120/K1100
YLA	MICROPLY SYNTACTIC FOAM	
NIPPON	XN - SERIES FIBERS	75/105/121 MSI



R. A. Lewis 16 June 93

#### CHARACTERIZATION

• OUTGASSING

MECHANICAL



R. A. Lewis16 June 93

#### POLYCYANATE OUTGASSING DATA ASTM E595

	TWI	NCM NCM	MATERIAL	SOURCE
•	.094%	000.	RS-3 (NEAT RESIN)	YLA
•	.20%	.01	954-3 (NEAT RESIN)	FIBERITE
•	%880.	000.	RS-3/XN50 (FV 69.9)	FORD AEROSPACE
•	.598%	.002	RS-3/MICROPLY/K139	TRW
•	%580.	.0003	RS-3/XN50	TRW
•	• 1.0%	0.1%	ACCEPTANCE VALUE	

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#### **MECHANICAL DATA**

- EQUIVALENT TO GR/EPOXY
- **USING FILMS AND PASTE EPOXY ADHESIVES** ACCEPTABLE BOND STRENGTHS

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#### PROCESSING ISSUES

- DEBULK
- · TACK
- VISCOSITY
- STORAGE
- OUTTIME
- MOISTURE ISSUES
- THIN TAPES



R. A. Lewis 16 June 93

## FABRICATION EXPERIENCE

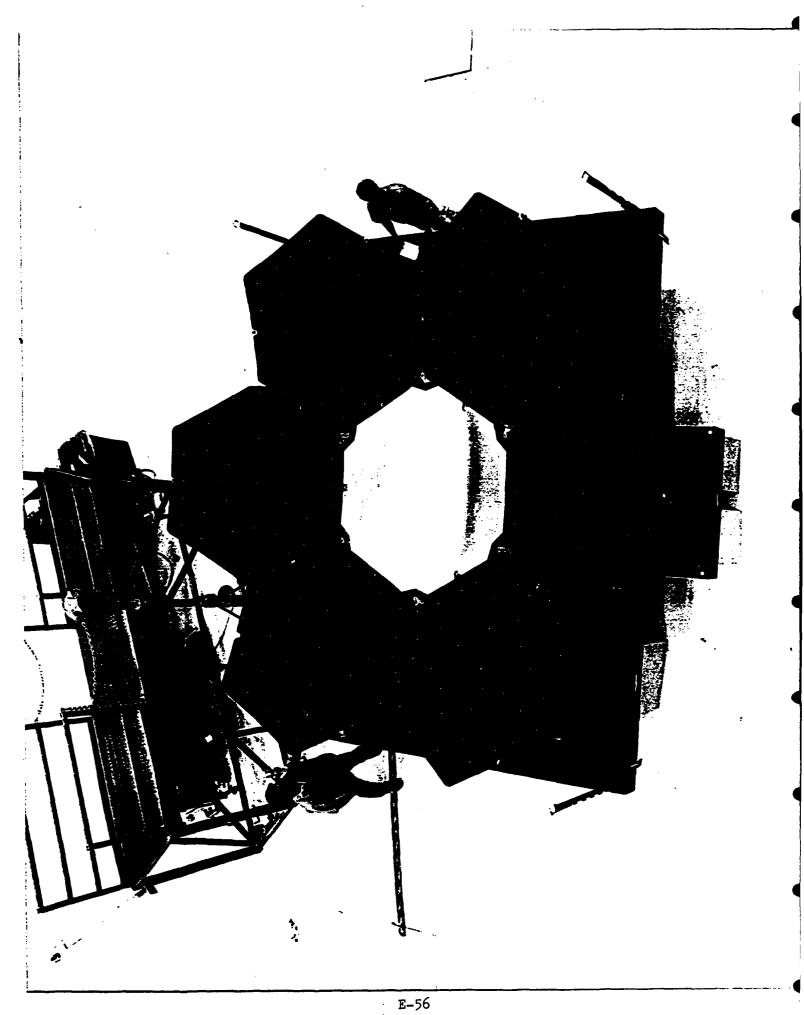
HARD

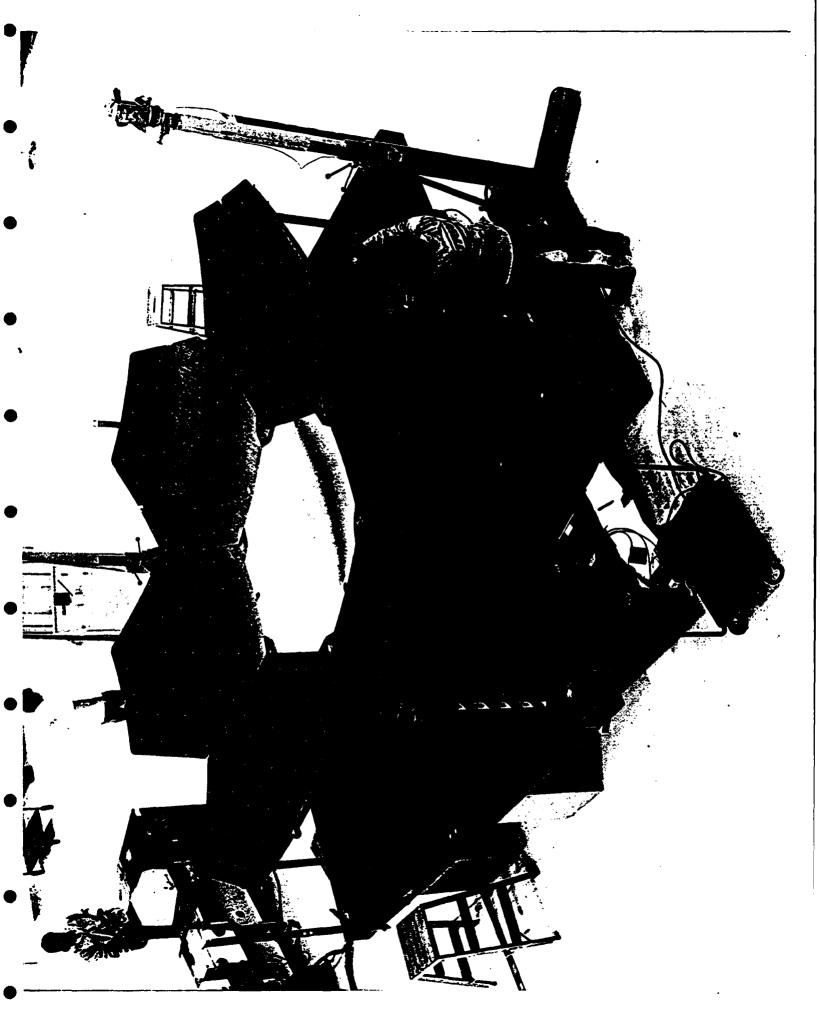
ULTRA THIN, 130 MSI PREPREG

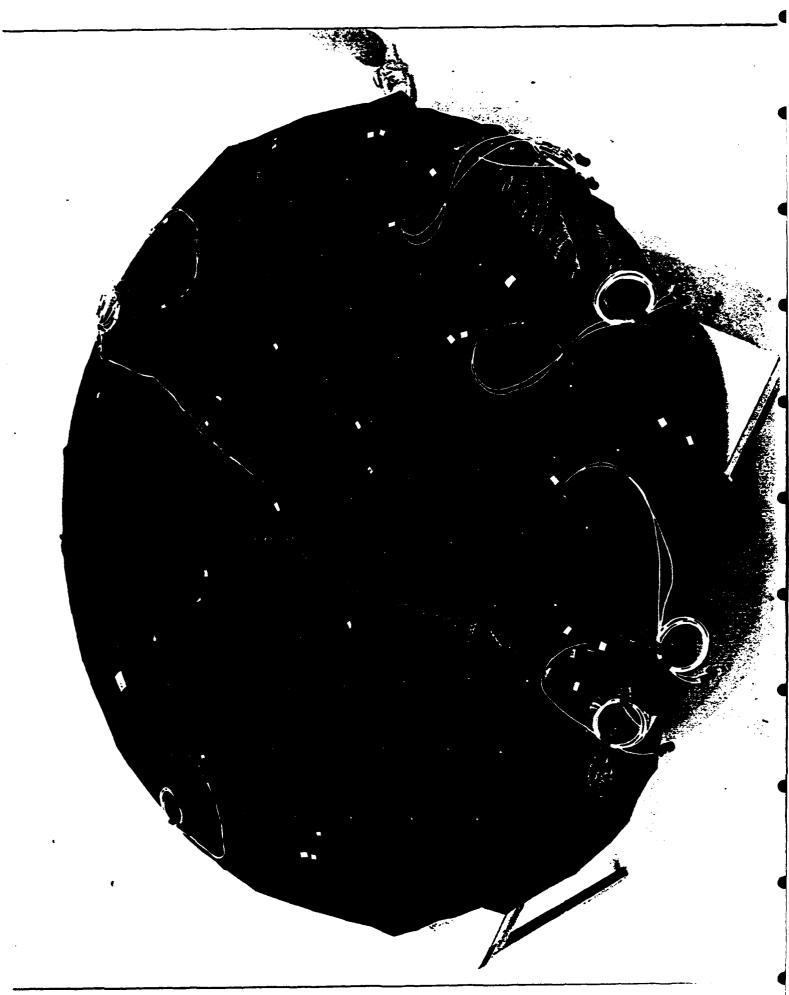
<1 MIL/PLY

THIN SANDWICH MEMBRANE

<15 MILS TOTAL THICKNESS







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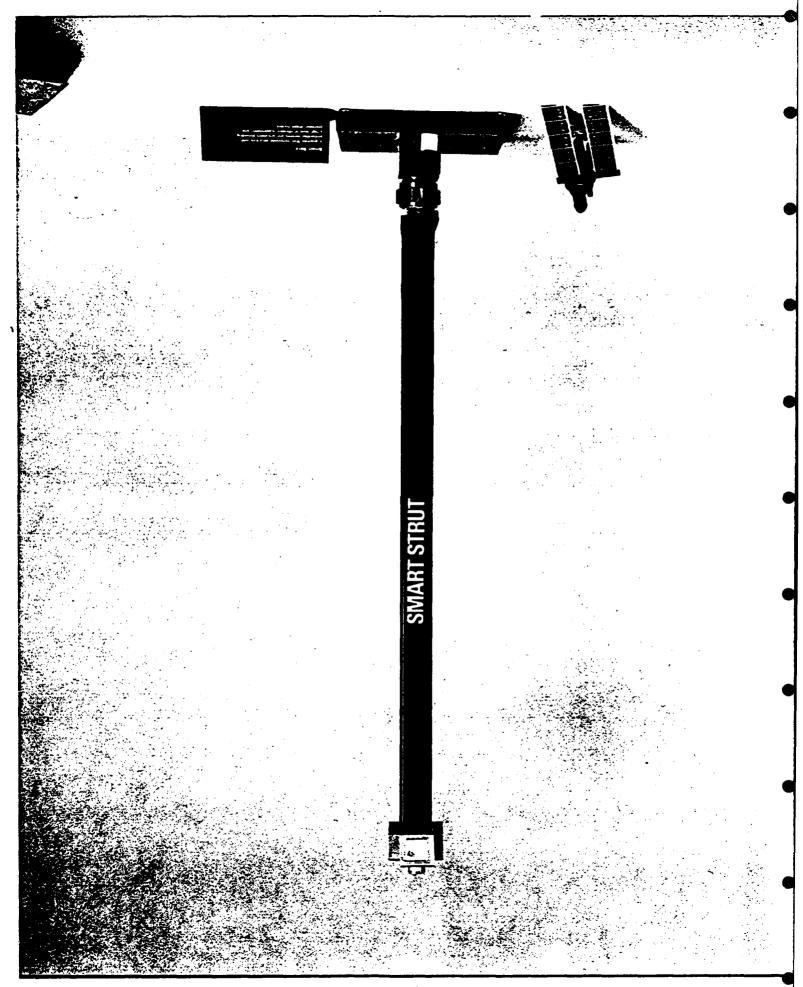
## FABRICATION EXPERIENCE

**AMASS** 

PRE-FLIGHT PHASE

ACTEX I

FLIGHT SCHEDULE FOR JULY 94





E-61



R. A. Lewis 16 June 93

#### ACTEX II

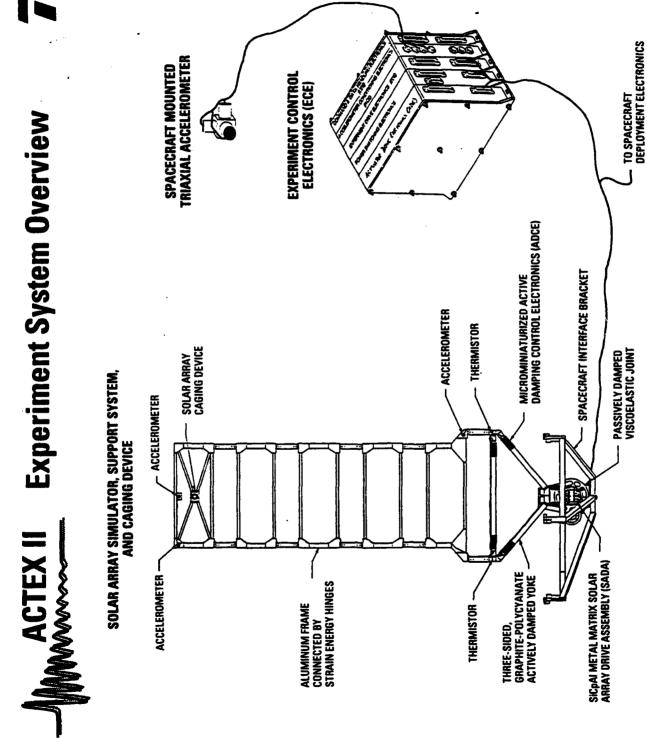
MATERIALS - T300/RS-3 TAPE

THICKNESS - 0.050 INCHES

LAYUP - HAND LAY UP

PROCESS - AUTOCLAVE (100 PSI, 350°F)

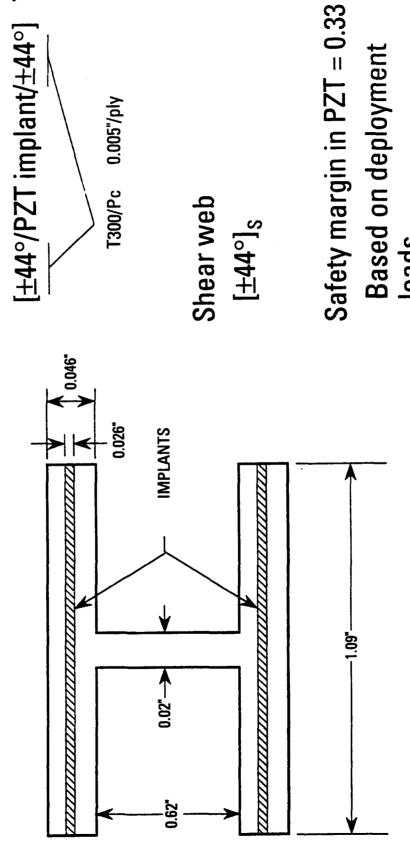






#### **Final Yoke Design**





Flange layup

loads

9300773 RG 004



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## FABRICATION EXPERIENCE

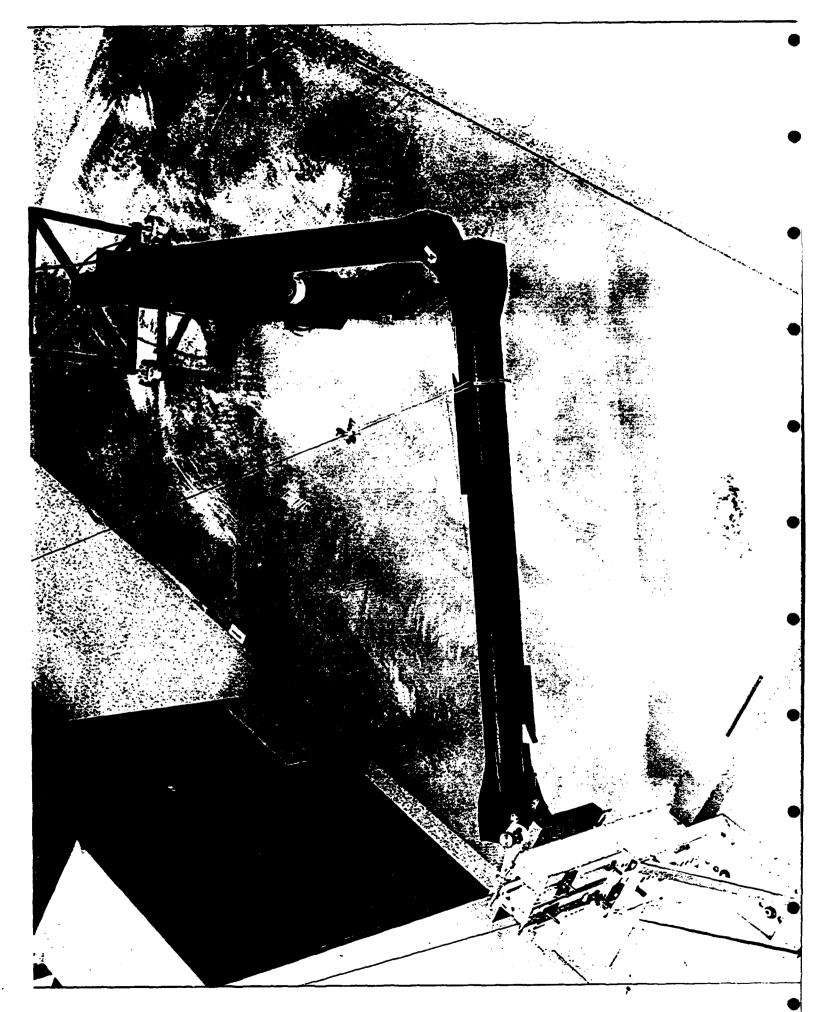
# INTEGRALLY HINGED SEGMENTED BOOM STRUCTURES

T50 FABRICS/RS-3

XN70A FABRICS/RS-3

XN70A TAPES/RS-3

MICROPLY FOAM/RS-3





R. A. Lewis 16 June 93

## FABRICATION EXPERIENCE

## SPACE EXPOSURE EXPERIMENTS

E01M - 3

**MATERIAL LAB** 

# Materials Experiments In Low Earth Orbit: Evaluation of Polycyanates (U)

- Significant effort is underway to evaluate polycanates for space applications
- Western Reserve University to conduct space environmental effects studies on polycyanate resins TRW is taking advantage of flight opportunities made available by the Strategic Defense Initiative Organization Technology Directorate (SDIO/TNK) and in collaboration with NASA and Case

Evaluation of Oxygen Interactions with Materials (EOIM-3)/STS-46 August 1993

- -- Comparative erosion yield study of RS-3 and PEEK graphite reinforced composites
- Preliminary results show that short duration tests at Shuttle altitudes produce sufficient surface recession to permit estimation of erosion yields.

Limited Duration Space Environment Candidate Materials Exposure LDCE-4.5 June 1993

- RS-3/XN50A [0,±45,0] 4-ply laminant
- Shuttle based experiment to measure in situ response of composite under mechanical stress to orbital atomic oxygen.

Matlab | October 1993

- "Free Flyer" deployed and retrieved by Shuttle. 45 hour ram exposure at 160 nm
  - Comparative exposure of seven 'cyanate formulations from competing vendors
    - -- measure surface recession to estimate erosion yield.
- Additional materials evaluation experiments are in planning to take advantage of space aboard SPAS-3 platform to be flown on STS-68 in late 1994.

# Cyanate Ester Technology at The Boeing Company

Cyanate Ester Workshop IDA, Incorporated Alexandria, Va June 16, 1993 Harry Dursch Lead Engineer Composites and Adhesives Group Boeing Defense & Space Group 206-773-0527

# Status of Cyanate Ester Technology at Boeing

ACESS Program and Preliminary Screening Results

Program description

Baseline design

Cyanate ester resin trade study results

Other Boeing Programs Using Graphite Reinforced Cyanate Esters

Boeing Defense & Space Group

## Cyanate Ester Fabrication Experience at Boeing

Material	Vendor	Years	Parts Built	Pounds Used
Modified RS-3 / Fiberglass	YLA	1990 - Present	Flat Laminates / Large Contour Shapes	1900
954-1 / 503 Quartz	Fiberite	1988 - 1991	Flat Laminates	200
954-3 / 503 Quartz	Fiberite	1990 - Present	Thin Flat Laminates / Large Thin Contour Shapes	200
2555 Film Adhesive	BASF / Narmco	1990 - Present	Secondarily Bonded Sandwich Parts	90
X-G 65 Bondsheet	Norplex - Oak	1987 - 1990	Thick Flat Laminates	200
954-2a / 581 and 503 Quartz	Fiberite	1993 -	26 Ft. Dia. Radomes (4th Qtr, 1993)	500

\* Process Development Usage

**Processes Like Epoxies** 

Boeing Defense & Space Group

#### All Composite Spacecraft Bus Structure (ACESS)

Customer: Phillips Laboratory / PKVC

(Naval Research Laboratory)

(SDIO / TNI)

Contract Number: ECP to F29601-92-C-0106

Contract Type: CPFF

Start Date: Mid June, 1993 (estimate)

Total Value: \$1.6M

Duration: 15 months

Subcontractor: Ball Aerospace

System Level Design Reqmts:

nts: Naval Research Laboratory

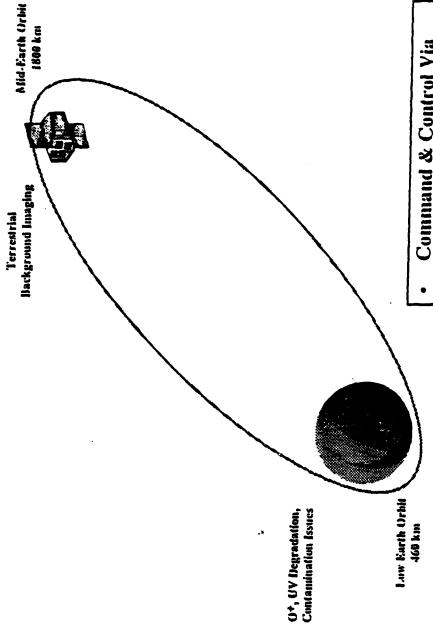
Design and build an all composite bus structure for the MDR / STRV2 space-Objective:

craft, to Naval Research Laboratory system requirements, for launch in July, testing. Demonstrate lower weight / cost and greater dimensional stability 1995 carrying U.K. and JPL experiments. Deliver to NRL for qualification than aluminum structure.

## STRV2 Mission Overview

#### STRV2 Technologies:

- MWIR Optical Sensor
  - Sensor (UK)
- Filters (SDIO)
- Baffles (SDIO)
- Cryocooler (SDIO or UK)
- Adaptive Structures (SDIO)
- Space Environment Experiments (SDIO)
- Advanced Materials & Structures (SDIO)

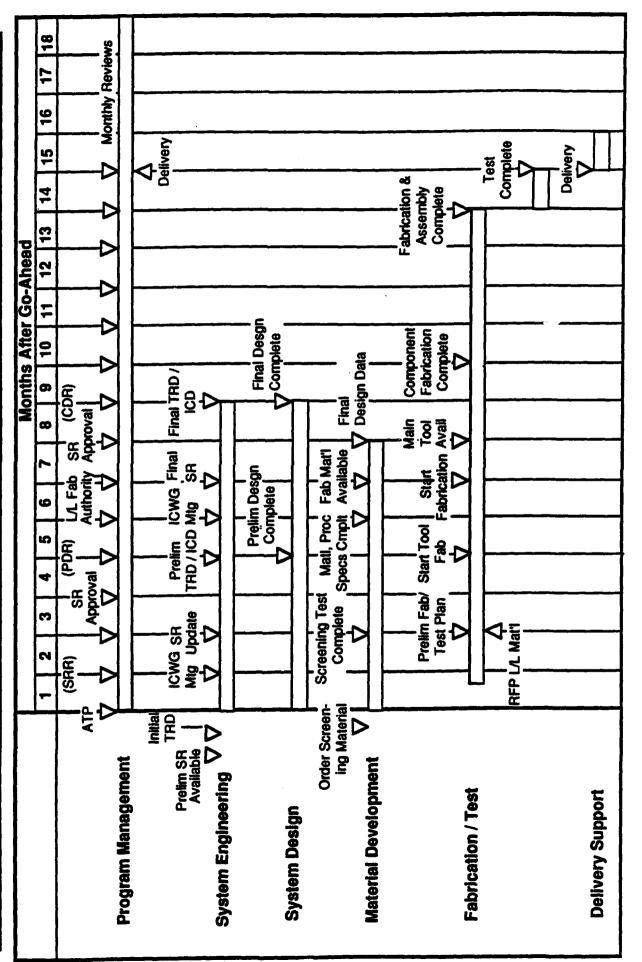


- Command & Control Via U.S. Assets; Data Collection Via U.S./UK Assets
  - Pegasus XL Launch; Sun Synchronous Orbit, 99.92° Inclination
- 1 Year Mission Life

Naval Research Laboratory Washington, DC

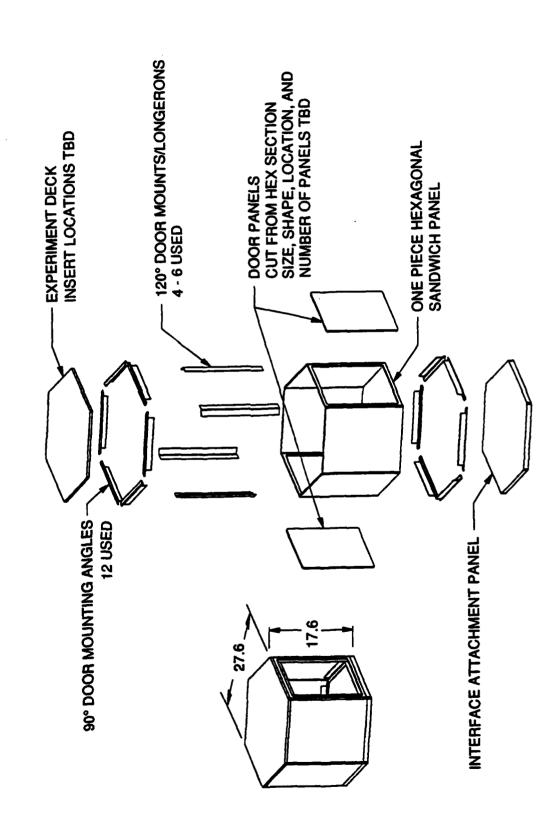


#### **ACESS Master Schedule**

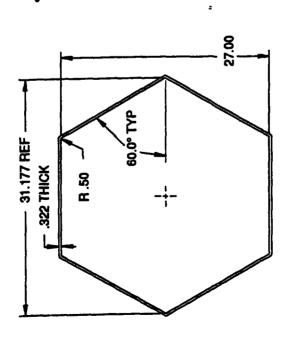


ACESS

#### Design Description Baseline Design



#### **Hex Section Details Design Description**

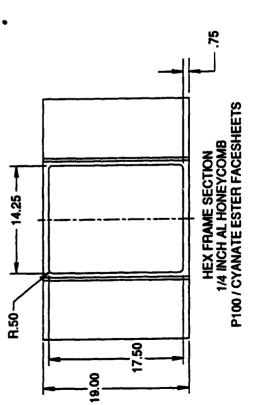




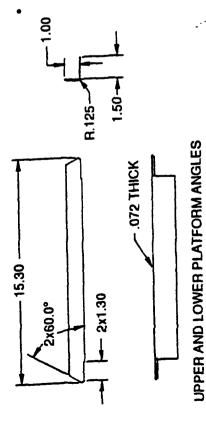
- Face sheets P100/cyanate ester 0.004" tape
- Lay-up optimized to give near-zero CTE longitudinally
- Aluminum honeycomb core 3.1 lbs/c.f. density x .25 inches thick
- flex-core, over-expanded
- Section layed up and cured at one time

#### **Door Panels**

- Cut from hex section
- Mount to secondarily bonded internal angles
- Design flexibility allows door panels to vary in size, shape, number, and location



#### **Design Description Component Details**

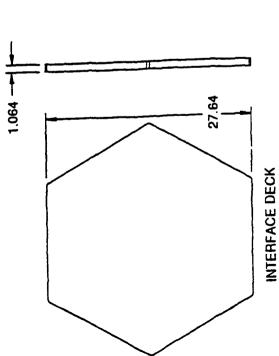


90° and 120° Angles

- 90 ° angles serve as both upper and lower platform mounts as well as horizontal door mounts
- 120 ° angles serve dual purpose
- Longeron stiffeners where needed
- Vertical door mounts
- Same lay-up used as hex section
- Higher modulus tape and/or fabric with cyanate ester

#### Experiment and Interface Decks

- Face sheets quasi-isotropic P100 tape lay-up
- Honeycomb core same as hex section except thickness
- Threaded and thru inserts pattern TBD



1 INCH AL HONEYCOMB WITH P100 / CYANATE ESTER FACESHEETS

ACESS WO Chart April 93 pp JOC 4



ACESS

### Co-Cured Composite Design Advantages

#### Reduced Part Count/Reduced Cost



Complex assembly tool no longer required

Co-curing reduces part count by ≥ 50 percent

Weight Savings Over Conventional Aluminum Bus

35 lbs total weight for aluminum without contingency

25 lbs total weight for composite with 15% contingency

Design Flexibility

Co-Cured (Baseline)

Insert locations not required immediately for design

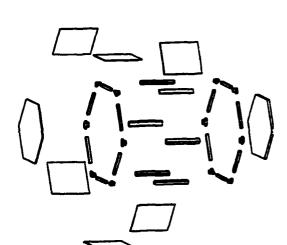
Panels size, shape, location, and number may vary

Tailorable laminates

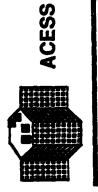
Facesheets can be made structurally stiffer and stronger

Thermal conductivity may be increased

Honeycomb core attributes may be varied



Secondary-Bonded



## **Material Requirements**

- Dimensional stability
- Mositure absorption, microcracking
- Low outgassing
- Low CTE
- Processability
- High quality prepreg
- · Co-curing honeycomb sandwich
- Material availability
- Thermal and electrical conductivity
- Strength and stiffness
- Space environment survivability

### **Neat Resin Materials**

	Ne	Neat Resin Material	-
Property	Cyanate Ester	Toughened Epoxy	Epoxy (934)
Moisture absorption Outgassing	1.5%	%0.9	7.2%
- TML - CVCM	0.01-0.07% 0-0.01%		
Microcracking	Improved	Improved	Baseline
Cure shrinkage	Positive	Negative	Negative
Tellabass	44 ppm/C		61 ppm/C
Glass transition	330 J/m² 128°C-182°C	219 J/m² 200°C	194°C
Flex modulus	0.40-0.44 Msi	0.55 Msi	0.6 Msi

# Cyanate Offer Greater Dimensional Stability and Improved Toughness

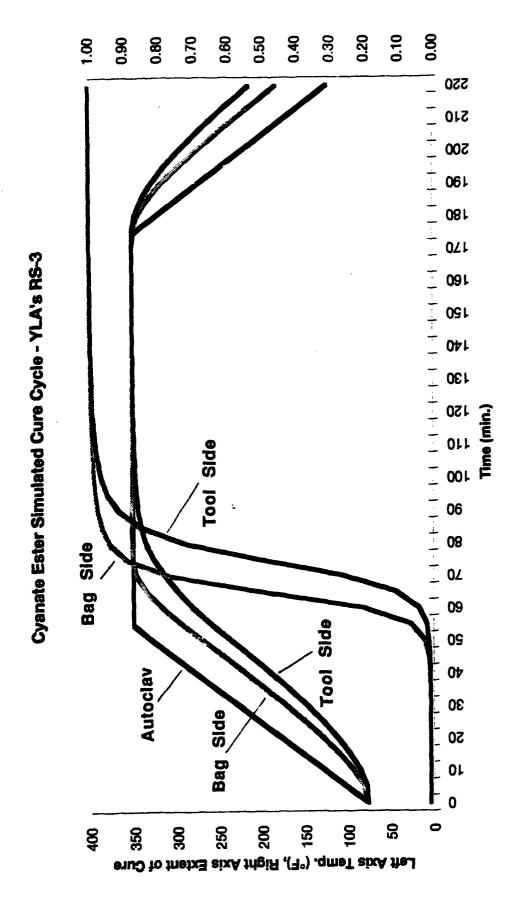
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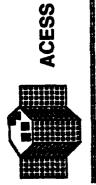
# Cyanate Ester Neat Resin Properties

		Near	Neat Resin	
Property	YLA	YLA	Fiberite	Bryte
	RS-3 (1)	RS-3M (1)	954-2a (1)	EX-1515 (2)
Outgassing				
- TML	0.07%	0.07%		0.01%
- CVCM	0.01%	0		0.01%
Glass transition	182°C	184°C		128°C
	244°C (3)	244°C (3)		
Viscosity (centipoise)	98	4401.	282	1983

(1) 2 hrs @ 350°F cure(2) 3 hrs @ 250° cure(3) 2 hrs @ 450 post-cure



0.00 0.10 0.70 0.50 0.40 0.30 0.20 1.00 0.90 0.80 0.60 Cyanate Ester Simulated Cure Cycle - Bryte's EX-1515 Tool Side 091 Side Bag Tool Autoclav 7 Left Axis Temp. (°F), Right Axis Extent of Cure



## M&P Development Approach

Early screening tests and trades to support material selection

Candidate materials for evaluation and trades

Cyanate ester: YLA RS-3 and RS-3M, Fiberite 954-2a, Bryte EX-1515

Toughened epoxies: Fiberite 977

Epoxy: Fiberite 934

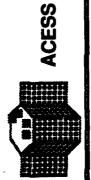
Fibers: P100, K1100, T650/42 fabric, IM7 fabric

Aluminum core: various thicknesses, cell sizes, and conformability

Develop program process spec and material spec

Material options available to minimize schedule risk

95514-abm-hd-93454.4



#### **Materials Summary**

Baseline is cyanate ester resin

Cyanate esters are "tougher" than toughened epoxies

Offer increased dimensional stability

- Offer low outgassing

- Quantities of high modulus fibers have been prepregged

- Processes like epoxies, with improved flow characteristics

Improved radiation hardness

Backup material is toughened epoxy

Fiberite's 977 family of resinsHercules 8552/8553

Short term availability of Dow Chemical-based cyanate ester TBD

Co-curing of cyanate ester/aluminum core into hexagonal shaped bus structure body represents moderate risk

Aluminum foil processing is existing technology at Boeing

## Additional Graphite-Reinforced Cyanate Ester Programs

- Pluto Fast Flyby
- JPL contract
- Design, build, and test 59" diameter ultra-lightweight duel frequency antenna
  - Cyante ester baseline. Dimensional stability in a controlled flow resin
- Integrated Power Panels
  - Phillips Lab
- Design, build, and test solar array panels
- Composite Structures Assembly
- Classified spacecraft program
- Design, build, and test flight hardware.
- Low Temperature Structural Carbon Fiber Prepreg
- Boeing Commercial evaluating 250°F cure systems including EX-1515

95514-abm-hd-93454.5

#### CJTD contract data sheet PP doc.

#### Research & Engineering

#### Technology Development Composite Joining

Boeing Defense & Space Group

Customer:

Phillips Laboratory

OLAC PL/VTSC

F29601-92-C-0106

Contract Number:

CPFF

Contract Type:

July 13, 1992

\$1.7M

Total Value:

Duration:

(39 Technical) 42 Months

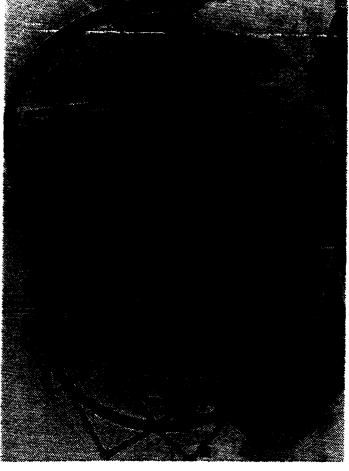
**Ball Space Systems Division** 

Subcontractors:

Objective:

Develop and demonstrate advanced polymeric composite joining improvements and promote the use of advanced composite technology that will provide system level performance

materials in Air Force surveillance/communications space systems.



Start Date:

#### Boeing Defense & Space Group

#### Summary

- Boeing Defense and Space Group experienced with design, fabrication and testing of cyanate ester based composites
- Radome applications
- Graphite reinforced systems
- Cyanate esters being selected for dimensional stablility and low outgassing in a controlled flow resin system
  - Process like epoxies
- Availability of Dow Chemical based cyanate ester resins is a concern

## **ULTRA HIGH MODULUS COMPOSITE STRUCTURES**

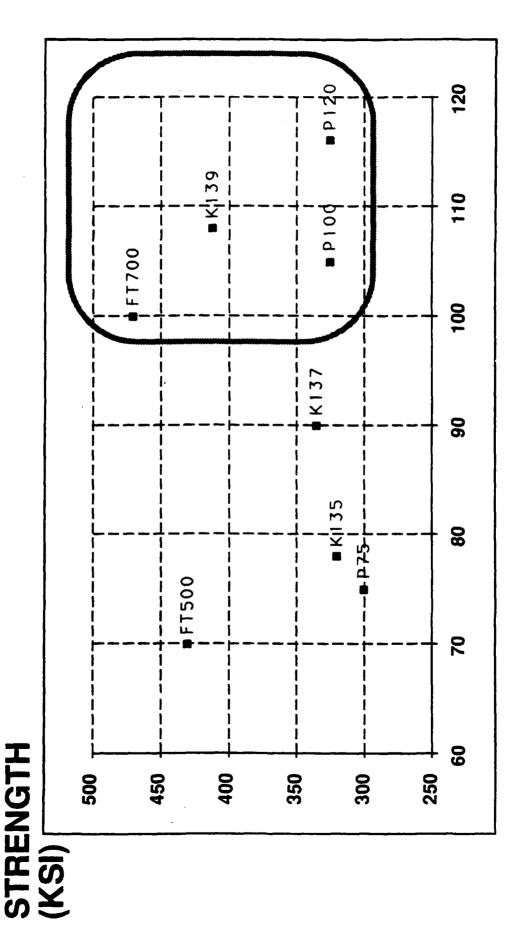
16 JUNE 1993

J. J. TRACY

MCDONNELL DOUGLAS AEROSPACE HUNTINGTON BEACH, CA

## FIBER TENSILE STRENGTH VS MODULUS

MCDONNELL DOUGL

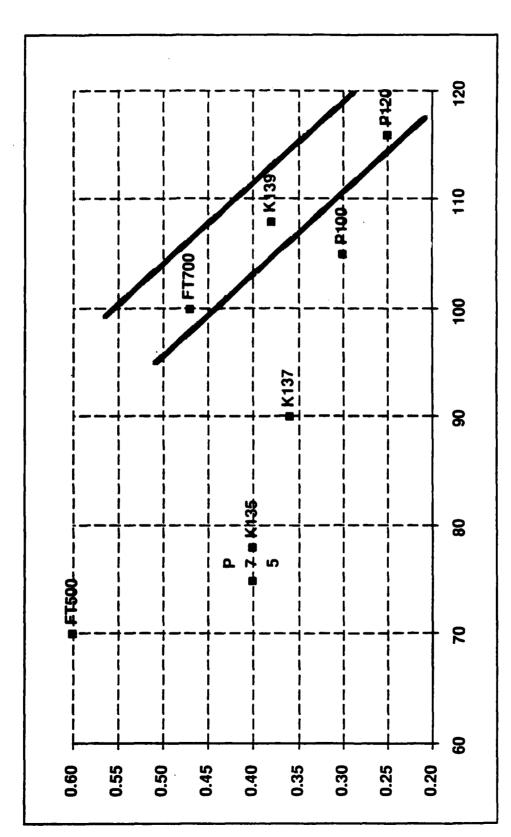


MODULUS (MSI)

## FIBER ELONGATION VS MODULUS

- MCDONNELL DOUG

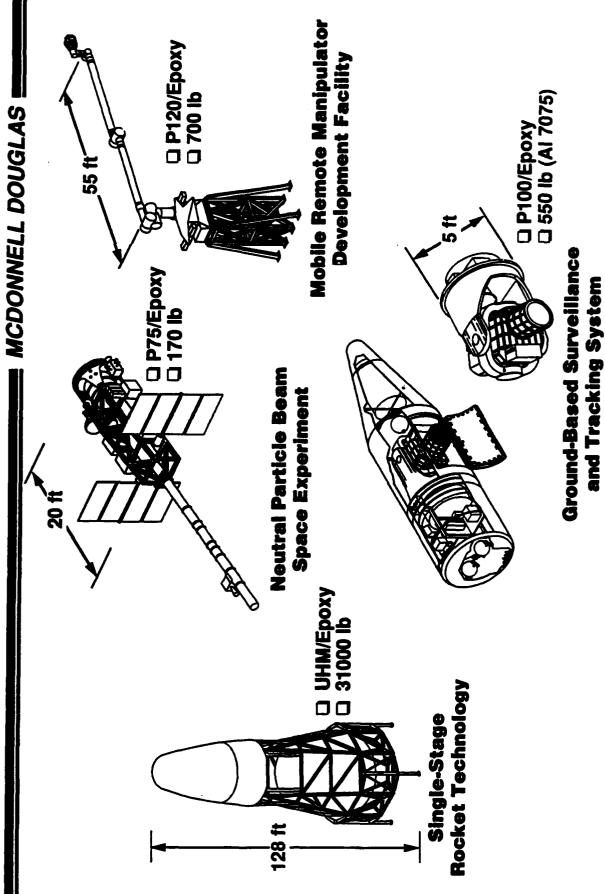


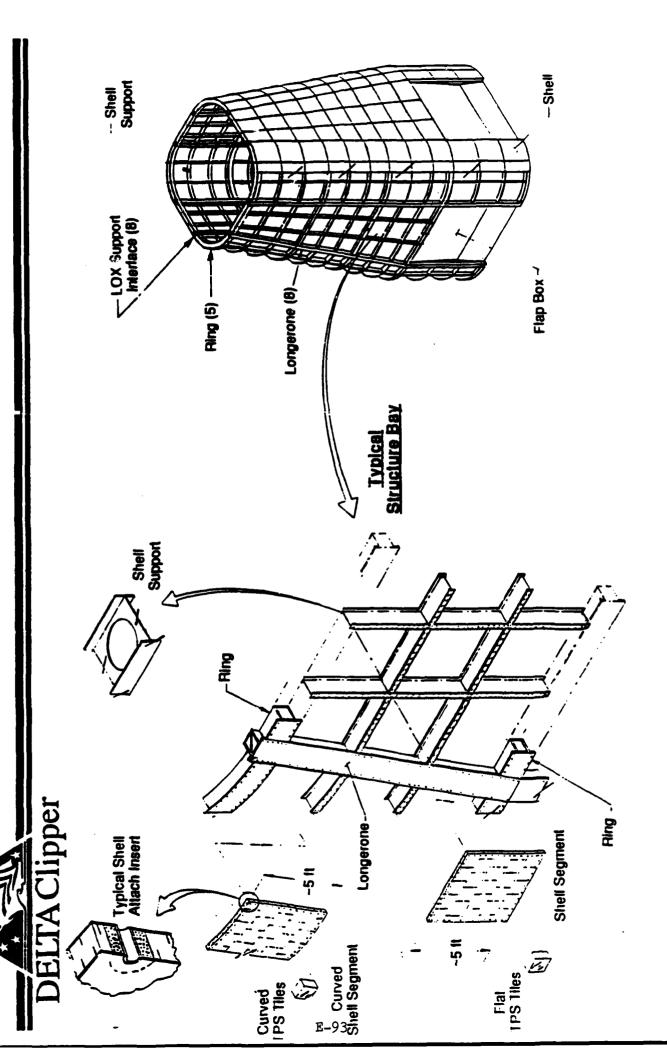


#### MODULUS (MSI)

# **EXAMPLES OF FUTURE SPACE SYSTEMS REQUIRING**VERY STIFF, LIGHTWEIGHT STRUCTURES

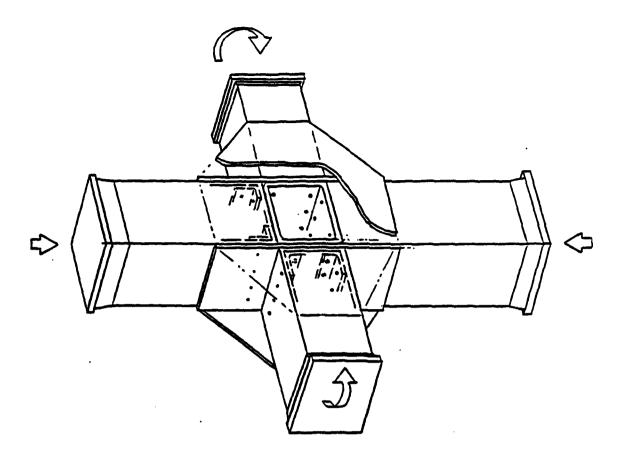
/CZ25177.2 M19DR





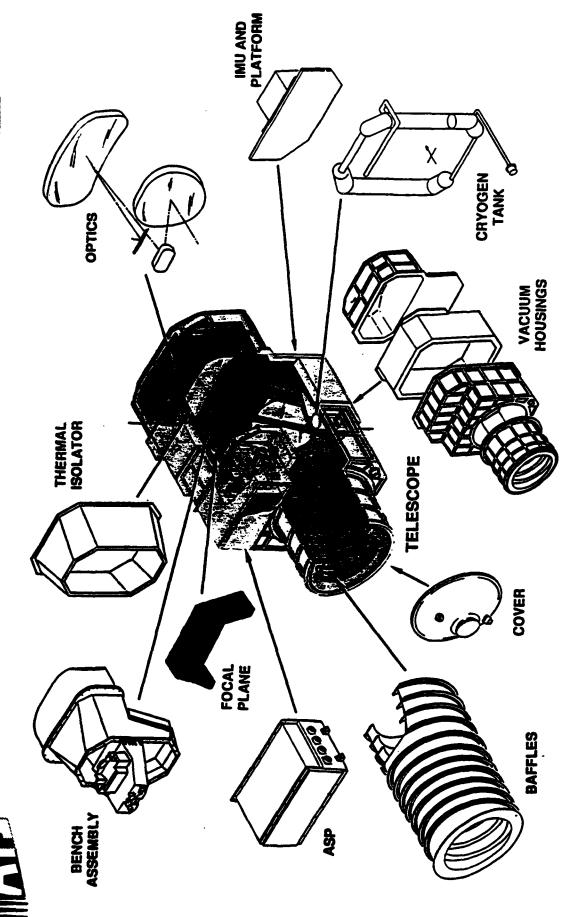
### TRUSS JOINT TEST: DESCRIPTION

- SINGLE TRUSS MEMBER
- APPLY ULTIMATE AXIAL COMPRESSION LOAD
- 8 STRAIN GAGES
- PURPOSE IS TO DETERMINE BASELINE PROPERTIES OF TRUSS MEMBER
- TRUSS JOINT
- APPLY ULTIMATE LOADS:
- CONSTANT BENDING MOMENT TO RING
- AXIAL COMPRESSION LOAD TO LONGERON
- 12 19 STRAIN GAGES
- PREDICTED FAILURE MODE IS LOCAL BUCKLING









- MCDONNELL DOUGLAS -

- HUGHES

CSA

### ADVANCED COMPOSITE GIMBAL

WCZB106.1 MITHE

#### ☐ Problem:

 Current sensor system has only 5% mass margin (34.8/690 kg); the gimbal is the heaviest component

#### ☐ Solution:

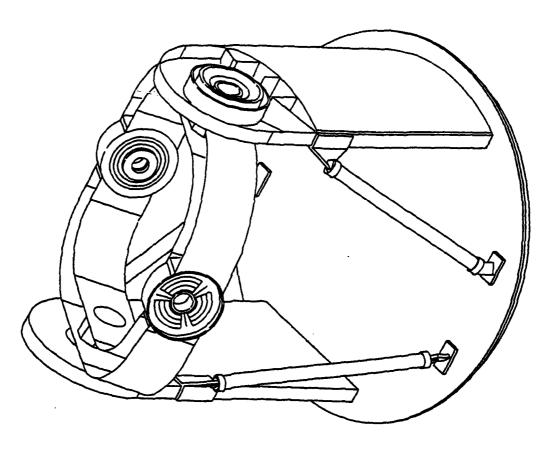
 Advanced composite gimbal structure weighing 30 to 40% less

#### ☐ Approach:

 Design, fabricate, and test high modulus composite ring, trunnions, base plate, and support struts

### Additional Benefits:

- Greater stiffness for improved servo margin
- Reduced motor torque and power
  - Reduced bearing loads
- Improved system growth potential
  - Classified benefits



### WEIGHT REDUCTION SUMMARY

MCDONNELL DOUGLAS

Component	Weight Reduction (Driven by 30 g Re-Entry Load)	Weight Reduction (Without Re-Entry; Driven by 10 g Launch Load)
Ring 19% Stiffer	16.2 kg (35%)	18.5 kg (40%)
Trunnions 81% Stiffer	23.0 kg (38%)	27.8 kg (46%)
Base Plate 3% Stiffer	19.4 kg (38%)	26.5 kg (52%)
Struts 37% Stiffer	8.2 kg (50%)	9.0 kg (55%)
Gimbal	66.8 kg (37%)	81.8 kg (45%)

## APPLICATIONS OF CYANATE ESTER RESIN ADVANTAGES TO MDA PROGRAMS

MCDONNELL DOUGLAS

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SS ANI	>			
SSANHONOL SONISS AD THO	>			`
		•	>	
		<i>&gt;</i>	•	
3411810WWOJ		`	`	
	SINGLE STAGE ROCKET TECHNOLOGY	NEUTRAL PARTICLE BEAM SPACE EXPERIMENT	GROUND BASED SURVEILLANCE AND TRACKING SYSTEM	MOBILE REMOTE MANIPULATOR DEVELOPMENT FACILITY

93D030/ov

# **ADVANTAGES OF CYANATE ESTER RESIN SYSTEMS**

MCDONNELL DOUGLAS

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$$(G_{1C} > 175 \text{ J/m}^2)$$

- High Toughness

- Low Dielectric Loss Tangent

## **CYANATE ESTER NEAT RESIN PROPERTIES**

---- MCDONNELL DOUGLAS ==

ONISS WOA				
1/0			0.01	0.000
SNISS VOLIO (30) THENSO			0.20	0.31
	1.30	1.24	1.19	1.19
AND ONLY OF THE OFFICE	194	215	206	254
MAN (S)	2.30	1.28	0.96	1.45
NINTH SENSITE ON STRAIN (SE) STRAIN STRAIN (SE) STRAIN STR	0.70	2.59	2.40	4.90
USA) HENSHIE WON I ENSIN	0.60	0.44	0.40	0.43
Marians	12.0	10.0	8.2	11.6
	Fiberite 934	Fiberite 954-2A	Fiberite 954-3	YLA RS-3

### LAMINATES TESTED

MCDONNELL DOUGLAS

Panel # 102/+4 2 102/+4 3 102/+4 4 102/+4 102/+4 102/+4	Layup			
102/4 3 102/4 102/4 102/4	AE/O/ AE/OT	Axial (0°) Fiber	Off-Axis Fiber	Resin
2 4 4 102/4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	S[0/c+-/0/c++/2o]	FT700	T800	034
3 [02/4 4 [02/4	$[0_2/+45/0/-45/0]_{\rm S}$	F1700	TROO	DEA 2
4 [0 <sub>2</sub> /+4	$[0_0/+45/0/-45/0]_{\rm e}$	0077	900 F	2-1-0
4 [02/+4		200	008	934
47./C	[ <sup>0</sup> 2/+45/0/-45/0]s	K139	T800	954-3
C++01	$[0/+45/02/-45/0]_{2s}$	P100	T650-42	<b>ERL</b> 1962
Panel #	Layup	Axial (0°) Fiber	Off-Axis Fiber	Door
50/745	10/445/90/-451			URAU
	// 00/-12/28 //00/-451	FT700	FT700	934
C++/0] /	[0/+45/80/-45]2s	F1700	F1700	954.3
8 [0/+45	$[0/+45/90/-45]_{2s}$	K139	K130	700
9 [0/+45	[0/+45/90/-45]2	K130		400
10 [0/+45	[0/+45/90/-45]	801	K139	954-3
	[0/. 4£/00/. 4£]	K13B	K13B	977-2
	/su/-45]2s	P120	P120	ERL 1939-3

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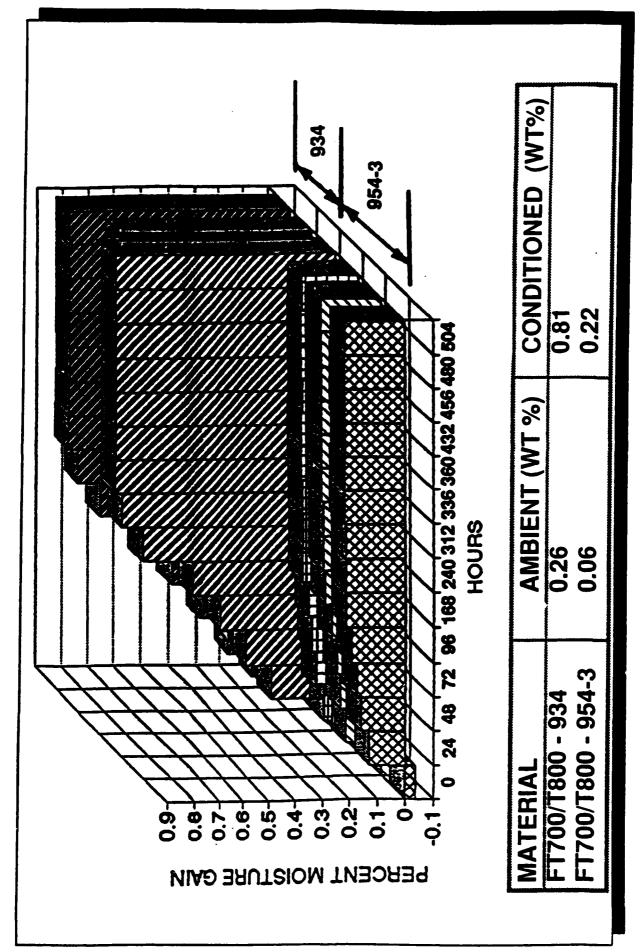
### **COUPON TESTS PERFORMED**

MCDONNELL DOUGLAS

Test Type	Properties	Specification	Comments
0° Tension	Ex, vxy, Fx (tension)	ASTM D3039	
0° Compression	Ex, vxy, Fx (compression)	SACMA SRM 1-88	Boeing-type specimen
In-Plane Shear	Gxy, Fxy	1	losipescu
Short-Beam Shear	Fx3	<b>ASTM D2344</b>	
Physical Properties	ν <b>f,Vv</b> , ρ	ASTM D3171 ASTM D792 ASTM D2734	
CTE	α×	<b>ASTM E 831-86</b>	-250F to +250F
Bolt Bearing	Fbr		Double lap

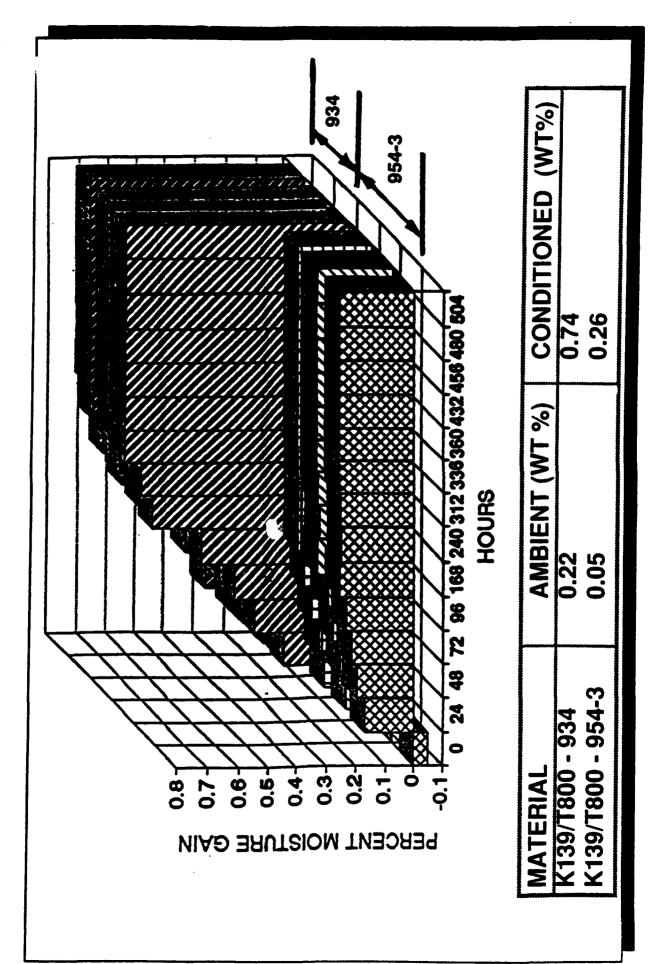
# PERCENT MOISTURE VS TIME - FT700

MCDONNELL DOUGLAS



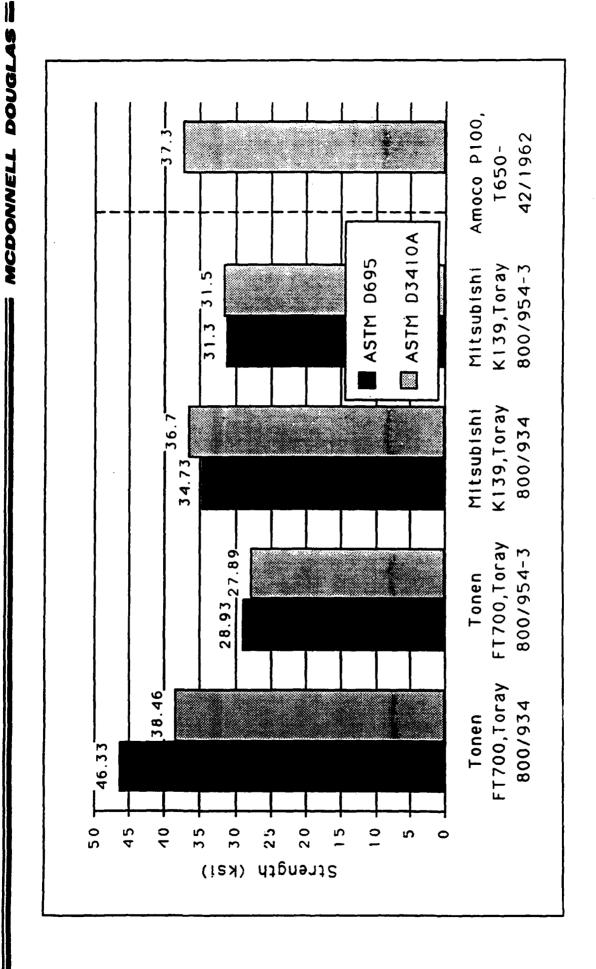
# PERCENT MOISTURE VS TIME - K139

= MCDONNELL DOUGLA



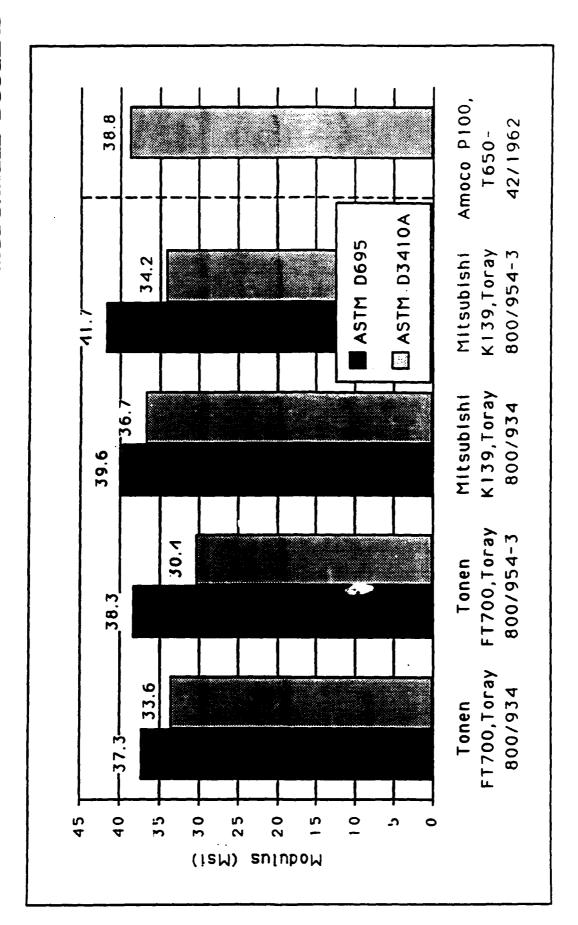
### **AMBIENT MOISTURE CONDITIONS**

# HYBRID LAMINATE COMPRESSION STRENGTH RESULTS

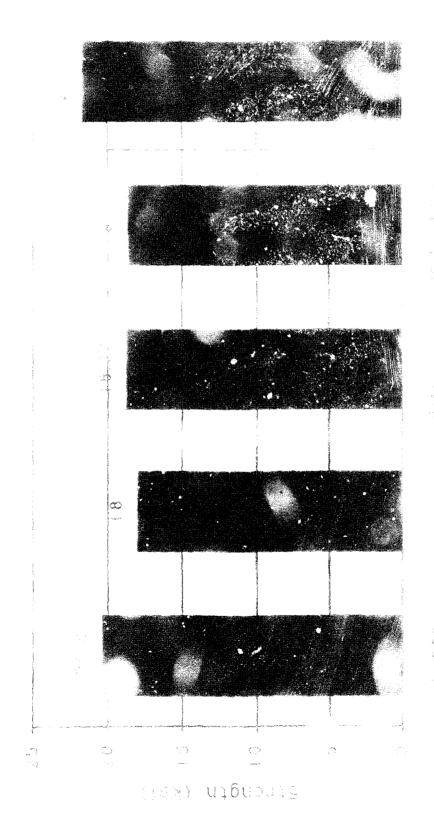


# HYBRID LAMINATE COMPRESSION MODULUS RESULTS

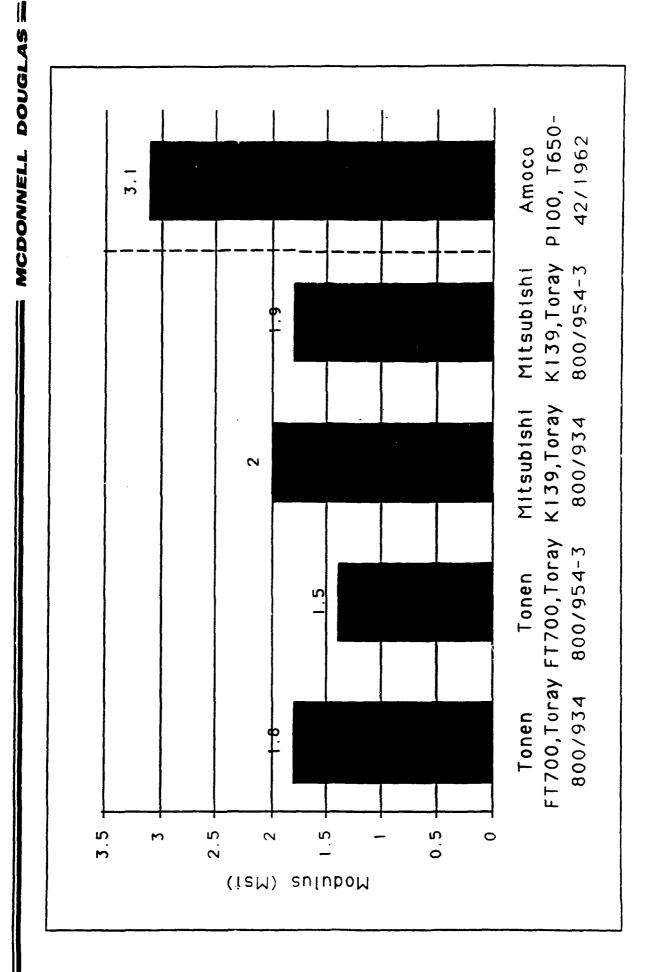
- MCDONNELL DOUGLAS



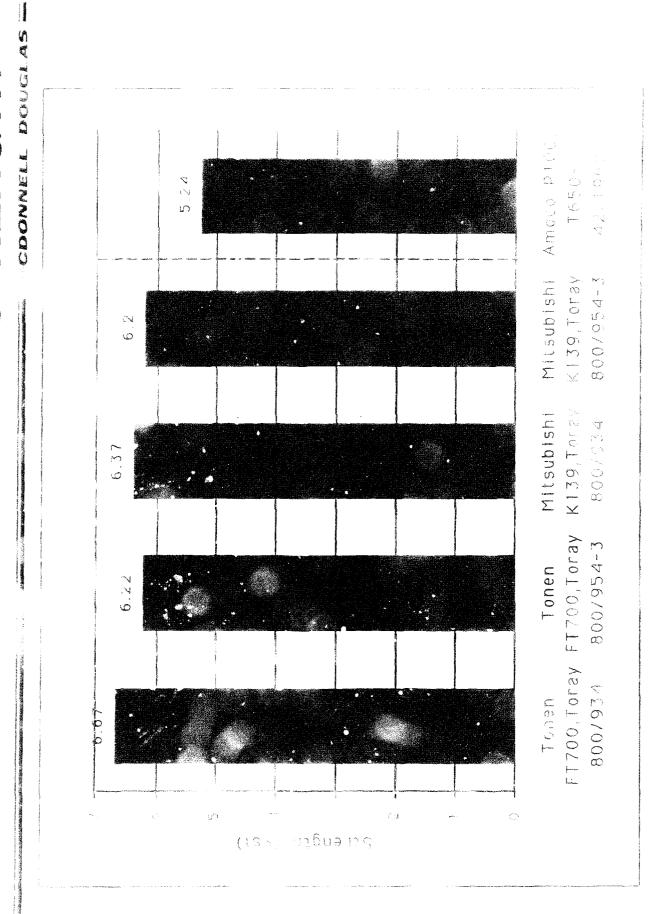
### **AMBIENT MOISTURE CONDITIONS**



## INPLANE SHEAR MODULUS



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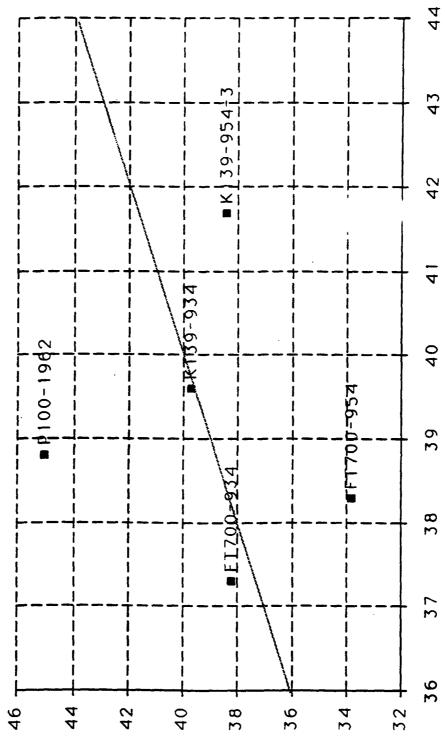


SHORT BEAM SHEAR, MOISTURE CONDUMENTED

# HYBRID LAMINATE TENSILE VS COMPRESSION MODULUS

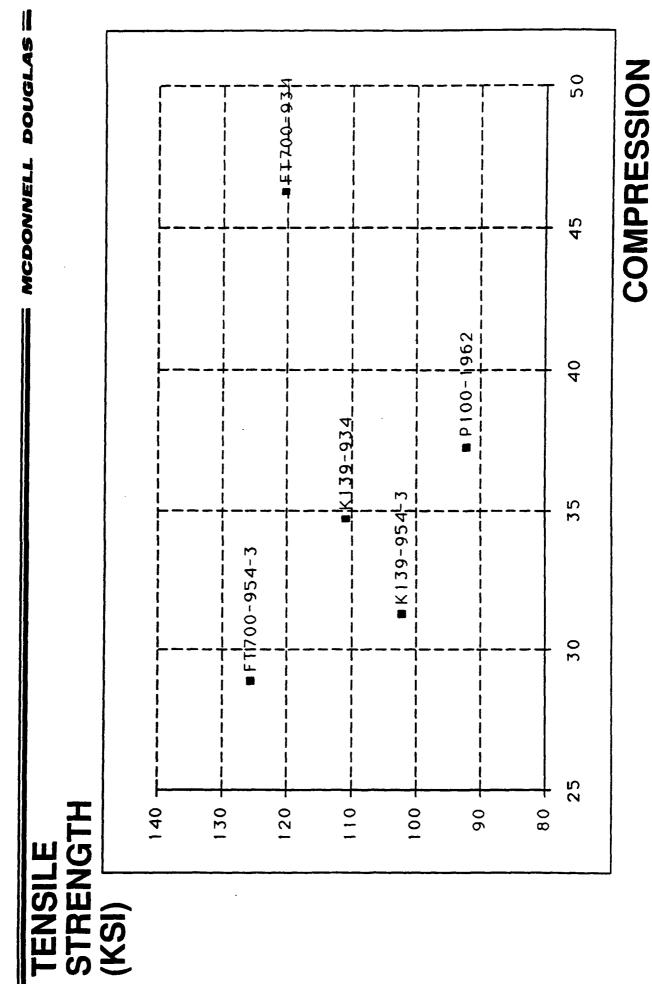
- MCDONNELL DOUGLAS

#### TENSILE MODULUS (MSI)



#### COMPRESSION MODULUS (MSI)

# HYBRID LAMINATE TENSILE VS COMPRESSION STRENGTH



STRENGTH (MSI)

## PSEUDO-ISOTROPIC LAMINATE TEST RESULTS

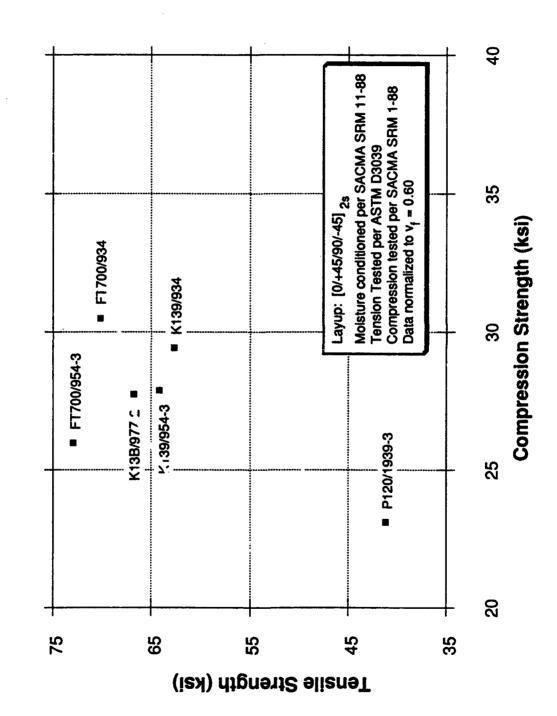
= MCDONNELL DOUGLAS ==

			Material	Material System		
Property	FT700/954-3	FT700/934	K139/934	K139/954-3	P120/1939-3	K13B/977-2
Tensile Strength (ksi)	72.89	70.25	62.72	64.16	41.15	62.99
Tensile Modulus (Msi)	20.70	21.02	23.09	21.91	23.12	23.87
Compression Strength (ksi)	25.97	30.50	29.43	27.85	23.12	27.70
Compression Modulus (Msi)	18.38	19.32	21.92	20.22	22.69	22.66
In-Plane Shear Strength (ksi)	18.90	19.08	18.99	19.49	15.16	17.41
In-Plane Shear Modulus (Msi)	6.33	6.25	6.94	7.22	6.22	7.45
Short Beam Shear Strength (ksl)	6.22	7.34	6.92	6.50	3.62	6.41
Ult. Bearing Strength (ksi)	64.7%	96.99	56.30	54.77	48.38	i

### PSEUDO-ISOTROPIC LAMINATE TENSILE vs. COMPRESSION STRENGTH

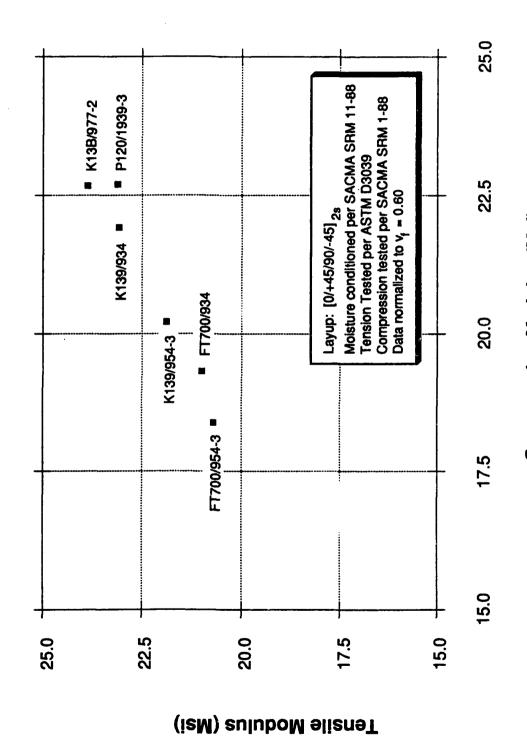
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MCDONNELL DOUGLAS



## PSEUDO-ISOTROPIC LAMINATE TENSILE vs. COMPRESSION MODULUS





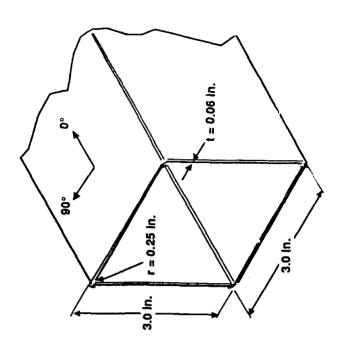
Compression Modulus (Msi)

### **UHM COMPOSITES R&D STUDY**

- MCDONNELL DOUGLAS

APROACH:

- □ COUPON TESTING
- HYBRID LAMINATES
- PSEUDO-ISOTROPIC LAMINATES
- ☐ FABRICATION STUDY
- HAND LAYUP
- FILAMENT WINDING
- ☐ TRUSS MEMBER COMPRESSION TESTING



**TRUSS MEMBER** 

### **TUBES FABRICATED**

MATERIAL	LAYUP	FABRICATION
P120/1939-3 IM7/8551-7A	[0 <sub>2</sub> /+45/-45/0 <sub>2</sub> ]sym	Hand layup
K139/977-2 AS4/3501	[0 <sub>2</sub> /+45/-45/0 <sub>2</sub> ]sym	Hand layup Filament wound
FT700/PRS	[0 <sub>2</sub> /+45/-45/0 <sub>2</sub> ]sym	Filament wound
FT700/934	[0 <sub>2</sub> /+45/-45/0 <sub>2</sub> ]sym	Hand layup
P75/1939-3	[0 <sub>2</sub> /+45/-45/0 <sub>2</sub> ]sym	Hand layup

### PROBLEMS ASSOCIATED WITH MANUFACTURING SQUARE CROSS SECTION TRUSS MEMBERS

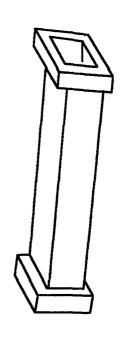
MCDONNELL DOUGLAS

- Low Viscosity Resin Resulted in Poor Quality Part
- Used Fiberite 954-3 Resin with Tonen FT700 Fiber
- 32% Resin Content in Prepreg
- 21% to 24% Resin Content in Cured Part
- Wrinkles in Part Caused by Loss of Resin
- □ Used Rubber Dams on Ends of Tooling

# POSSIBLE SOLUTIONS TO MANUFACTURING PROBLEMS CAUSED BY LOW RESIN VISCOSITY

MCDONNELL DOUGLAS

- Use Resin with Added Thickening Agents
- Use Tooling with Integral Dams



 Use Cure Cycles with Reduced or No Autoclave **Pressure** 

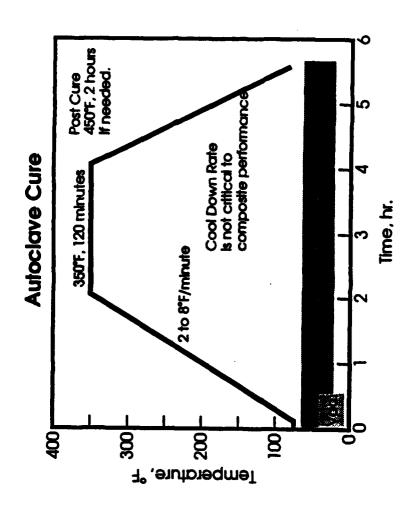
### VISCOSITY TRADE-OFFS

- ☐ Fiberite 954-2A has higher viscosity than 954-3
- Improve resin flow control during cure
- ☐ Drawbacks of 954-2A versus 954-3
- Slightly higher moisture pickup due to thermoplastic additives (still 3 times better than epoxy)
- More difficult to prepreg
- ☐ Laminates made with 954-2A will have equivalent mechanical properties compared to laminates made with 954-3

# RECOMMENDED YLA RS-3 AUTOCLAVE CURE CYCLE

MCDONNELL DOUGLAS

- Apply vacuum on part at room temperature
- Apply 45 to 80 psi
- Release vacuum
- Heat part to 350°F (+10°F/-0°F)
- Hold at 350°F for 120 Min (+15 Min/-0 min)
- Cool part, release pressure, and debag



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### SUMMARY OF CYANATE ESTER RESINS **VERSUS EPOXY RESINS**

MCDONNELL DOUGLAS

### ☐ ADVANTAGES

- Lower Moisture Uptake
- Lower Outgassing
- **Better Dimensional Stability**
- **Higher Toughness**
- **Higher Tg**
- **Lower Dielectric Loss Tangent**
- Lower Elelectric Constant

### ☐ DISADVANTAGES

- Lower Compression Strength
- Lower Compression Modulus
- Low Viscosity

#### Form Approved REPORT DOCUMENTATION PAGE OMB No. 0704-0188 minor of information is nationated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and 3. REPORT TYPE AND DATES COVERED . AGENCY USE ONLY (Leave blank) 2. REPORT DATE November 1993 Final--June 1993-December 1993 4. TITLE AND SUBTITLE 5. FUNDING NUMBERS C - MDA 903 89 C 0003 Graphite-Reinforced Polycyanate Composites for Space and Missile Applications T- T-R2-597.09 **AUTHOR(S)** Janet M. Sater, Michael Rigdon 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) 8. PERFORMING ORGANIZATION REPORT NUMBER Institute for Defense Analyses 1801 N. Beauregard St. IDA Document D-1454 Alexandria, VA 22311-1772 SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(EE) 10. SPONSORING/MONITORING AGENCY REPORT NUMBER **Bailistic Missile Defense Organization** The Pentagon Washington, DC 20301-7100 11. SUPPLEMENTARY NOTES 12a. DISTRIBUTION/AVAILABILITY STATEMENT 12b. DISTRIBUTION CODE Approved for public release; distribution unlimited. 13. ABSTRACT (Maximum 180 words) LtCol Michael Obal of the BMDO Materials and Stuctures Office sponsored this workshop to promote communications among the participating groups and to identify technical issues and limitations associated with graphite-reinforced polycyanate composites. Some of the identified issues include a lack of standard materials and associated process specifications due to continuing changes in resin and prepeg formulations. Perhaps the most discussed issue was the lack of a statistical database, due, in part, to the changing formulations just mentioned. Development of a handbook via government coordination of existing information may be one way to provide data to designers and increase material acceptance. In addition, high resin contents in prepeg tapes; a lack of appropriate protective coatings; a need for structural bonding processes; a need for additional, environment-related data, particularly effects on mechanical/physical properties; a need for joint tests and subsystem level tests of structural components; and limited design tools and standards for using composites were discussed. 15. NUMBER OF PAGES 14. SUBJECT TERMS structures, spacecraft, composite missile structures, continuous fiber-reinforced 467 polymer composites, cyanate ester resins, high modulus graphite fibers, BMDO 16. PRICE CODE Materials and Structures 19. SECURITY CLASSIFICATION 20. LIMITATION OF ABSTRACT 17. SECURITY CLASSIFICATION 18. SECURITY CLASSIFICATION

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